# MICELLAR CATALYSIS OF THE HYDROLYSIS OF ARYLTRIFLUOROACETATES

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## ABSTRACT

The neutral hydrolysis of aryl trifluoroacetates was investigated in micellar media of cetyl trimethylammonium bromide (CTAB) and sodium dodecylsulphate (SDS). The reaction is catalyzed by both CTAB and SDS micelles, with larger rate enhancement in CTAB. The reaction mechanism ($B_{AC}3$) is the same in both micellar media and their pseudophases as in non-micellar medium .The Hammett s-r relationship is obeyed with reaction constants ( ) of +2.46 in non-micellar medium, +2.21 in CTAB, +2.30 in SDS micellar media and +1.79 and +2.18 in CTAB and SDS pseudophases, respectively.

## INTRODUCTION

Micellar catalysis of organic reactions has been extensively studied over the years (Fendler and Fendler, 1975; Khan, 2007)). Interest in this area comes mainly from the fact that being interface reactions, they can be considered as models for enzymatic catalysis. Following the earlier findings from Menger and Portnoy (1967) and subsequent studies by others (Cordes, 1973; Romsted, 1977, 1984), it is now generally agreed that the magnitude of the observed micellar catalysis of a nucleophilic reaction depends, among other factors, on the relative rate of reaction in the aqueous pseudophase ($k_2^w$) and the micellar pseudophase ($k_2^m$).

It has also been shown that the orientation of the substrate molecules in the surfactant aggregate affects the relative reaction rates in the aqueous and micellar pseudophases (Broxton *et al.*, 1989). For a quantitative treatment of these reactions, the pseudophase ion exchange model is widely accepted although this model breaks down for some types of surfactants (Romsted, 1984).

Nucleophilic reactions in organic systems are known to be very susceptible to solvent effects, and in some cases, not only do the reaction rates change from one solvent system to another, the actual mechanism of reaction changes (Talbot, 1972). One of the most widely accepted methods of investigating if a change in mechanism of a reaction occurs is structure-reactivity study leading to the determination of Hammett reaction constants (Hammett, 1970). In fact, this methodology has also been used in mechanistic enzymology.

Reactions in micellar pseudophase can be considered, in a way, as being in a different solvent system. In this study, we investigated the hydrolysis of aryl trifluoroacetates in the presence of micelles of cetyltrimethylammonium bromide (CTAB) and sodium dodecylsulphate (SDS) (Equation 1).

$$CF_3 - \overset{\overset{\displaystyle O}{\displaystyle \|}}{C} - OAr + H_2O \longrightarrow CF_3COOH + ArOH \quad \dots \dots \dots \dots [1]$$
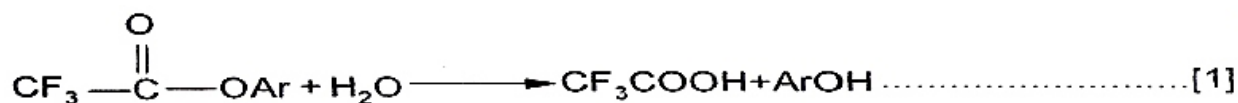
Using the Hammett relationship, we show that the reaction follows the same mechanism in both aqueous and micellar media.

## MATERIALS AND METHODS
### Materials

Phenyl trifluoroacetate and the substituted phenyl trifluoroacetates (I) were prepared from trifluoroacetic anhydride (0.15 mole) and the corresponding phenols (0.10 mole) using the method of Sakakibara and Inukai (1965). A mixture of the trifluoroacetic anhydride and each of the phenolic compounds was refluxed for about 5 hours until all the crystals disappeared. The resulting solution was then concentrated to dryness to give the crude product. Trifluoroacetic anhydride (Aldrich), acetonitrile (Fisher Scientific), cetyltrimethylammonium bromide (CTAB) (Aldrich) and sodium dodecylsulphate (SDS) (Aldrich) were all  used without further purification. The esters were purified by distillation except 4-nitrophenyl trifluoroacetate which was recrystallized from hexane.

I
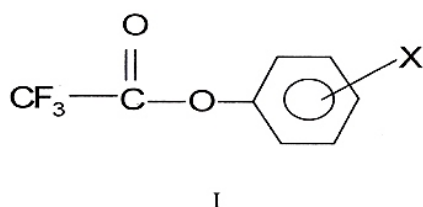
X: H, 3-Cl, 4-Cl, 3-NO₂, 4NO₂, 3-CH₃, 4-CH₃

The measured physical constants of the substituted phenyl trifluoroacetates with the respective literature values (Neuvonen, 1986) are presented below:

·   X=H, boiling point 119-121°C; literature boiling point 123°C
·   X=3-Cl, boiling point 177-179°C; literature boiling point 177-178°C
·   X=4-Cl, boiling point 184-185°C; literature boiling point 183°C
·   X = 3-CH₃, boiling point 122-124°C
·   X=4-CH₃, boiling point 126-127°C
·   X = 3-NO₂, melting point 40-43°C literature melting point 41-41.6°C
·   X=4-NO₂, melting point 37-38°C literature melting point 36-38°C

**Preparation of Solutions**

The stock solution ($10^{-3}$ mol dm$^{-3}$) of each of the esters (Fendler and Fendler 1975) was prepared by dissolving the mass of the appropriate ester in acetonitrile inside a 25cm³ volumetric flask and making it up to meniscus mark with the solvent.

The aqueous CTAB stock solution ($5.0 \times 10^{-3}$ mol dm$^{-3}$) was prepared by dissolving 0.046g of CTAB in distilled water in a 25 cm³ standard flask at room temperature. Similarly, the aqueous SDS stock

solution ($5.0 \times 10^{-3}$ mol dm$^{-3}$) was prepared by dissolving 0.360g of SDS in distilled water inside a 25cm³ standard flask and the solution made up to mark at room temperature. The required concentrations of esters and surfactants (CTAB and SDS) were prepared from the stocks by making appropriate dilutions.

**Kinetics**

The reaction rates were followed by monitoring the increase in absorbance of the phenol released at appropriate absorption maximum, $_{max}$[270 nm (H), 282 nm (3-Cl), 290 nm(4-Cl), 333nm (3-NO₂), 310nm (4-NO₂), 273nm (3-CH₃) and 274nm (4-CH₃)] as a function of time using a SP-400 UV-Visible Pye-Unicam Spectrophotometer with a thermostated cell holder. The required volumes of water and surfactant were mixed and allowed to reach thermal equilibrium (25°C) in a cuvette (!cm path length) contained in the jacketed cell compartment of the UV-Visible Spectrophotometer. The required volume of the trifluoroacetate solution in acetonitrile was then added to initiate the reaction.

The hydrolysis rate coefficient ($k_{obs}$) for each kinetic run was obtained from the slope of the linear plot of $\ln(A_t-A_8)$ versus time where $A_8$ is absorbance at infinite time and $A_t$ is absorbance at time t. The $k_{obs}$ values were also calculated by the method of Guggenheim (Frost and Pearson, 1961) and found to agree with those from linear plots.

The second order hydrolysis rate constant $k_w$ (mol$^{-1}$ dm$^3$ s$^{-1}$) for each of the esters was calculated from equation 2:

$$k_w = \frac{k_{obs}(micelle - free)}{[H_2O]} \quad \dots\dots\dots\dots\dots\dots\dots\dots[2]$$

**RESULTS AND DISCUSSION**

The first-order rate constants and the activation parameters of hydrolysis of the aryl trifluoroacetates ($3 \times 10^{-4}$ mol dm$^{-3}$) in non-micellar medium are presented in Table 1.

This reaction has been studied earlier in the region 5.56-38.9M (water in acetonitrile), in which a general base-catalyzed mechanism (scheme 1) was proposed (Neuvonen, 1986).
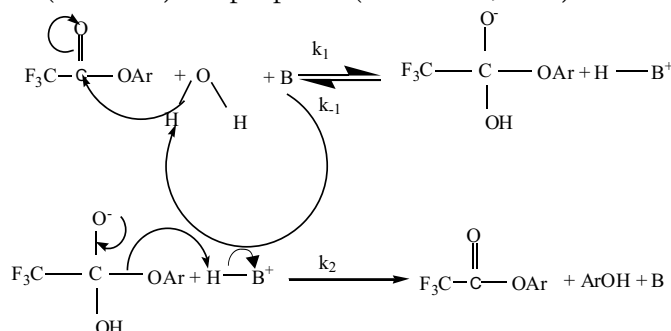


Scheme 1

B in this case was considered to be another water molecule acting as a catalyst. The transition state for the rate limiting step was given as Scheme II.



**II**
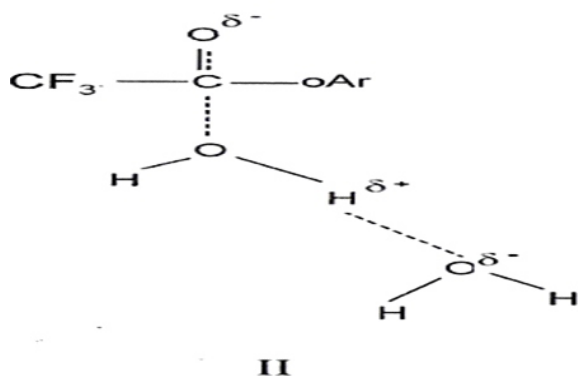
Results obtained in this study are consistent with the same mechanism. Large negative entropies of activation are observed for all the ester reactions (Table 1). The difference in absolute values of our activation parameters and those of Neuvonen (1986), $\Delta H^* = 13.1 \, \text{kJmol}^{-1}$, $\Delta S^* = -165 \, \text{Jmol}^{-1}\text{K}^{-1}$ for 4-nitrophenyl trifluoroacetate in 0.56M $H_2O$ is consistent with differences in the extent of solvation of the initial and transition states under the two conditions.

The observed rate constants correlate well with the substituent constants ($\sigma_x$), giving a reaction constant $\rho$ value of +2.46 (Fig. 1) which is almost identical with Neuvonen's value of +2.44 for the same reactions in 0.56-3.89 M (water in acetonitrile) (Neuvonen, 1986).

**Table 1: Rate Constants and Activation Parameters ($\Delta H^*$ and $\Delta S^*$) of the Hydrolysis of Substituted Phenyl Trifluoroacetates ($3.00 \times 10^{-4}$ mol dm$^{-3}$) in Non-Micellar Medium.**

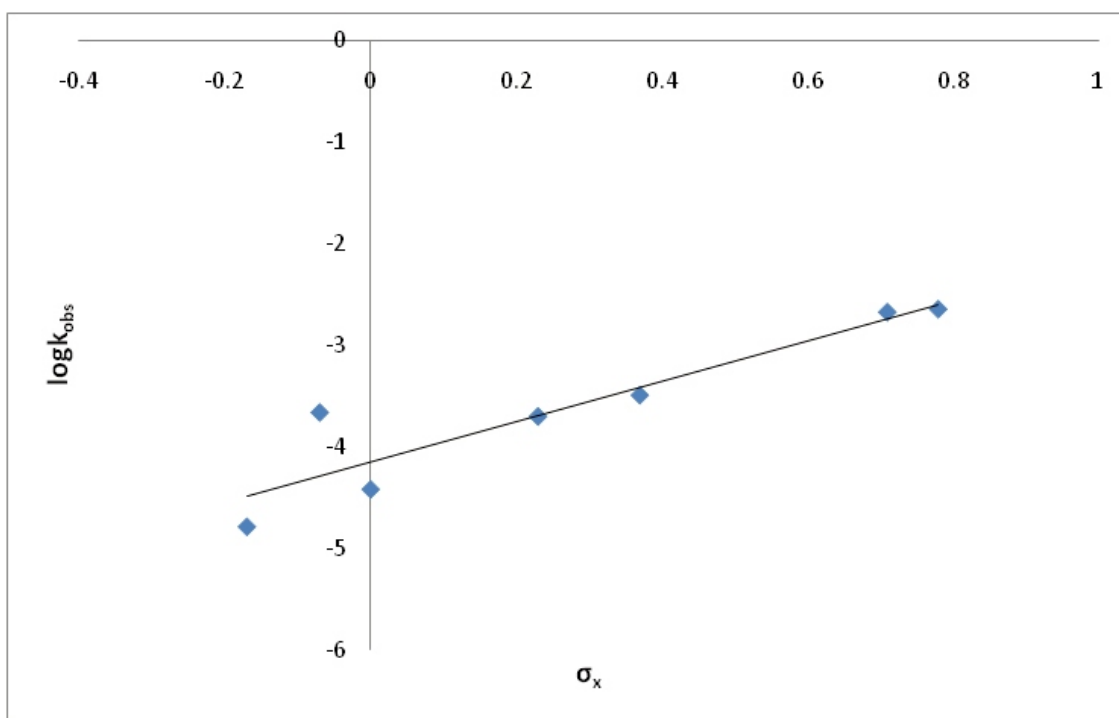| Phenyl substituent X | $k_{obs}$ ($\times 10^4$) (s$^{-1}$) at 25$^\text{O}$C | $\Delta H^*$(kJmol$^{-1}$) | $\Delta S^*$(Jmol$^{-1}$K$^{-1}$) | log$k_{obs}$ | $\sigma_x$ |
|---|---|---|---|---|---|
| H | 0.38±0.01 | +22.0± 0.5 | -237± 3 | -4.42 | 0.00 |
| 3-Cl | 3.22±0.02 | +30.3 ± 0.1 | -210 ± 3 | -3.49 | 0.37 |
| 4-Cl | 2.00 ± 0.01 | +28.7 ± 0.2 | -220± 1 | -3.70 | 0.23 |
| 3-NO₂ | 21.4 ±0.01 | +26.4± 0.2 | -213 ± 1 | -2.67 | 0.71 |
| 4-NO₂ | 22.80 ±0.02 | +32.3 0± 0.2 | -196 ± 1 | -2.64 | 0.78 |
| 3-CH₃ | 0.22 ±0.01 | +42.8 ± 0.1 | -171 ± 1 | -3.66 | -0.07 |
| 4-CH₃ | 0.16 ±0.02 | +22.7 ± 0.2 | -242 ± 2 | -4.79 | -0.17 |



**Fig. 1: Hammett-Type log k$_{obs}$ Vs Substituent Constants ($\sigma_x$) Plot for Substituted Phenyl Trifluoroacetates Hydrolysis in Non-Micellar Medium at 25$^\circ$C.**

## Hydrolysis in CTAB Medium

The $k_{obs}$-CTAB concentration profiles for the hydrolysis of the esters are shown in Fig. 2. The profiles show a progressive rate increase up to a maximum. This observation is the consistent pattern generally associated with micelle-catalyzed bimolecular reactions (Dunlap and Cordes, 1968;

Zakharova *et al.*, 2001). The observed catalysis suggests that the trifluoroacetates are solubilized in a region where there is sufficient concentration of water molecules and with increased activity relative to the bulk water region.

From temperature-dependence studies using constant ester and CTAB concentrations, we also
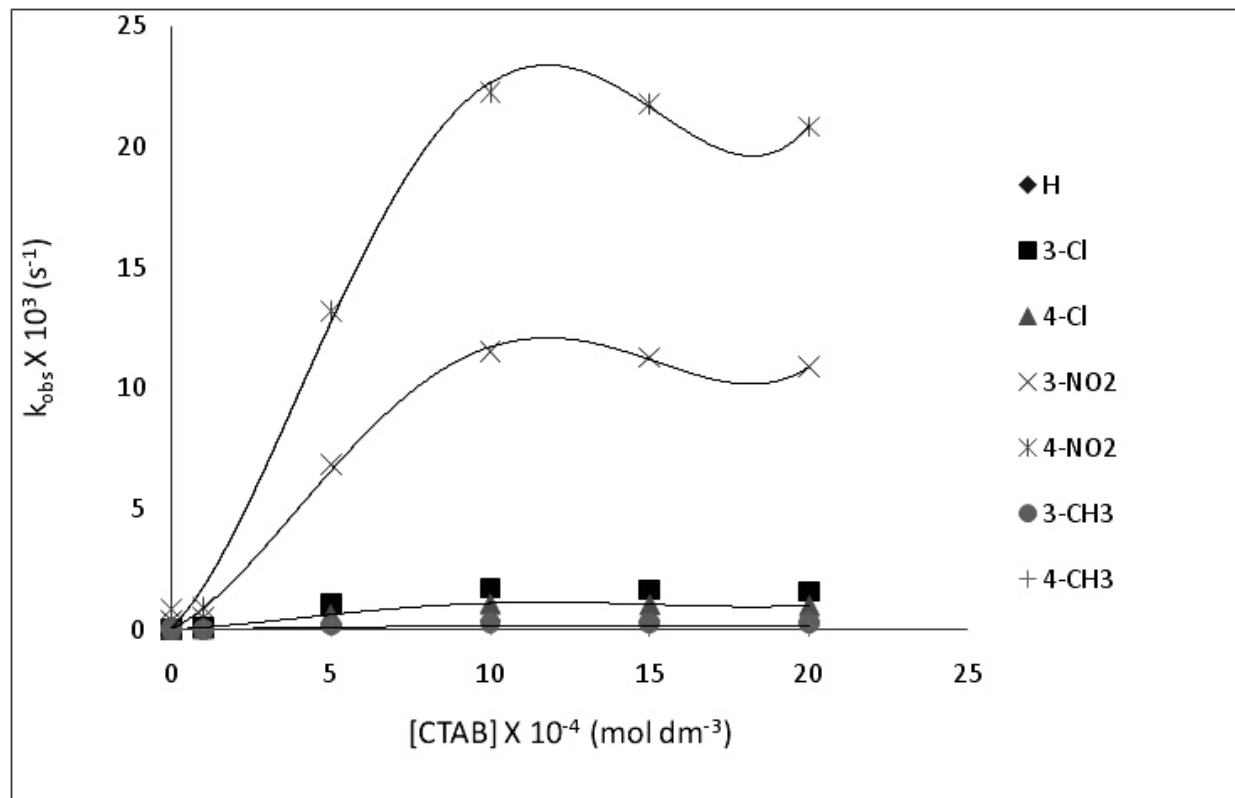
**Figure 2: Variation of $k_{obs}$ with [CTAB] at Fixed Substituted Phenyl Trifluoroacetate Concentration ($5.0 \times 10^{-4}$ mol dm$^{-3}$) at 25°C.**

**Table 2: Activation Parameters ($\Delta$H* and $\Delta$S*) in the Hydrolysis of Fixed Substituted Phenyl Trifluoacetate Concentration ($5.0 \times 10^{-3}$ mol dm$^{-3}$) and Fixed CTAB Concentration ($1.0 \times 10^{-3}$ mol dm$^{-3}$)**

| Phenyl substituent X | $k_{obs}$ $\times 10^4$ (s$^{-1}$) at 25°C | $\Delta$H*(kJmol$^{-1}$) | $\Delta$S*(Jmol$^{-1}$K$^{-1}$) | log $k_{obs}$ | $\sigma_x$ |
|---|---|---|---|---|---|
| H | 3.99 ±0.01 | +22.9± 0.2 | -210 ± 2 | -3.40 | 0.00 |
| 3-Cl | 17.33 ±0.02 | +27.7 ± 0.1 | -190 ± 2 | --2.76 | 0.37 |
| 4-Cl | 1.08 ±0.02 | +27.8 ± 0.1 | -195 ± 2 | -2.97 | 0.23 |
| 3-NO$_2$ | 115.39 ±0.02 | +27.8± 0.1 | -182 ± 2 | -1.94 | 0.71 |
| 4-NO$_2$ | 222.99 ±0.03 | +30.2 ± 0.5 | -175 ± 2 | -1.65 | 0.78 |
| 3-CH$_3$ | 3.16 ±0.01 | +27.8 ± 0.2 | -195 ± 2 | -3.50 | -0.07 |
| 4-CH$_3$ | 1.78 ±0.02 | +22.7± 0.2 | -215 ± 2 | -3.75 | -0.17 |

Figure 3 is the Hammett plot of the reactions in CTAB. A linear relationship is obtained with a reaction constant ( ) of +2.21. That the Hammett relationship is followed by the micelle-catalyzed reaction and with a similar reaction constant as in

non-micellar medium implies that the hydrolysis of aryl trifluororacetates responds to substituents in the same fashion for both uncatalyzed and CTAB-catalyzed situations, suggesting that the reaction mechanism is the same in both media.

The implication of this is that the reaction could not be occurring in the micellar core where there are very few water molecules. This slightly lower value of the reaction constant in CTAB is consistent with the reactivity- selectivity principle (Hirsch, 1974). The reactions, being faster in CTAB medium, are less susceptible to substituent effects than in non-micellar medium.
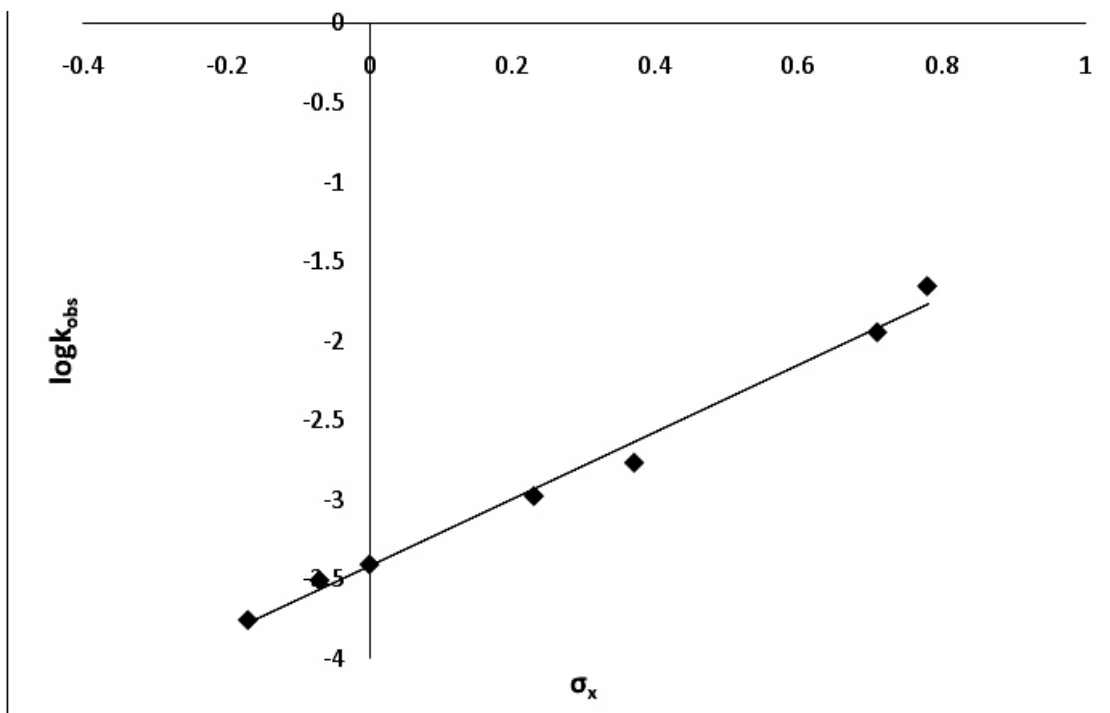


**Figure 3 : Hammett-Type log $k_{obs}$ vs Substituent Constants ($\sigma_x$) Plot at Fixed Concentration(5.0 X $10^{-4}$ mol dm$^{-3}$) of Substituted Phenyl Trifluoroacetates and Fixed CTAB concentration(1.0 X $10^{-3}$ mol dm$^{-3}$) at 25°C.**

Using the pseudophase model (Shinoda and Hutchison, 1962), the second-order rate constant in the micellar pseudophase ($k_2^m$) was calculated for each hydrolysis in CTAB (Table 3) using equations 3 - 5:

$$k_2^m = 0.14 \, k_M \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots [3]$$

$$k_M = k_{obs} \text{ (in micelles ) } \times \frac{[D_n]}{[H_2 0]} \dots\dots\dots\dots\dots\dots [4]$$

and $(D_n) = [D] - CMC\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots.[5]$

where $[D_n]$ = concentration of micellized CTAB

$[D]$ = Stoichiometric concentration of CTAB

CMC  Critical micelle concentration of CTAB

Table 3: Second Order Rate Constants ($k_w$, $k_M$, $k_2^m$) at fixed CTAB Concentration (1.0 x $10^{-3}$ mol dm$^{-3}$) and fixed Concentration (5.0 x $10^{-3}$ mol dm$^{-3}$) of Substituted Phenyl Trifluoroacetates at 25°C.

| Phenyl substituent X | $k_w$ (x$10^6$) mol$^{-1}$dm$^3$s$^{-1}$ | $k_M$ (x$10^8$) mol$^{-1}$dm$^3$s$^{-1}$ | $k_2^m$(x$10^9$) mol$^{-1}$ dm$^3$s$^{-1}$ | log $k_2^m$ | $\sigma_x$ |
|---|---|---|---|---|---|
| H | 0.25 | 1.4 | 2.0 | -8.70 | 0.00 |
| 3-CI | 2.20 | 6.2 | 8.7 | -8.06 | 0.37 |
| 4-CI | 1.37 | 3.0 | 5.4 | -8.27 | 0.23 |
| 3-NO$_2$ | 14.65 | 41.6 | 58.2 | -7.24 | 0.71 |
| 4-NO$_2$ | 30.06 | 80.0 | 112.4 | -6.95 | 0.78 |
| 3-CH$_3$ | 0.18 | 1.1 | 1.6 | -8.80 | -0.07 |
| 4-CH$_3$ | 0.11 | 0.6 | 0.9 | -9.05 | -0.17 |

A plot of log $k_2^m$ versus $\sigma_x$ is presented in Figure 4. The reaction constant $\rho$ is +1.79. This lower value of $\rho$ vis-à-vis that obtained in the entire CTAB micellar medium suggests that the reaction is less susceptible to subsituent effects in the micellar pseudophase than in the entire micellar medium or non-micellar medium.

The linear relationship obtained in the log $k_2^m$ versus $\sigma_x$ plot coupled with a reaction constant of positive sign evaluated therefrom shows that the reaction mechanism in the micellar pseudophase is not significantly different from that operating in the entire micellar medium. The linear relationship obtained in the log $k_2^m$ versus $\sigma_x$ plot implies that the reaction obeys the Hammett relationship, in the CTAB micellar pseudophase, as in non-micellar medium. This demonstrates the applicability of the Hammett relationship to reactions occurring in micellar psuedophase. In addition the positive sign of the reaction constant in the micellar pseudophase which is the same as in non-micellar medium, also implies that the reaction mechanism is retained in CTAB micellar pseudophase, as in non-micellar medium, the significant rate difference in the two media notwithstanding.
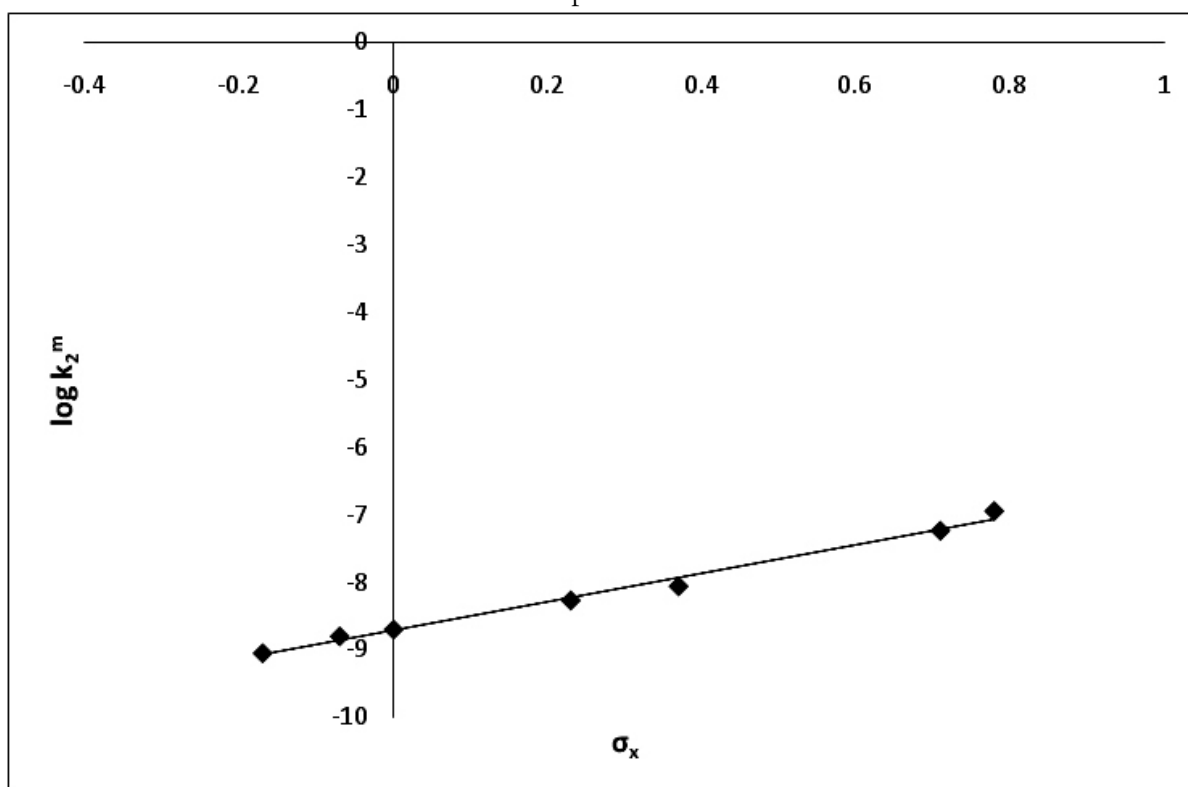


**Figure 4: Hammett-Type log $k_2^m$ vs Substituent Constants ($\sigma_x$) at Fixed Concentration(5.0 X 10$^{-4}$ mol dm$^{-3}$) of Substituted Phenyl Trifluoroacetates and Fixed CTAB Concentration (1.0 X 10$^{-3}$**

### Hydrolysis in SDS Medium

The $k_{obs}$-surfactant concentration profiles for the hydrolysis of the esters in the presence of SDS are shown in Figure 5. As in CTAB, the profiles follow the general pattern of micellar effects on bimolecular reactions. The catalysis also suggests that the esters are solubilized in a region of the micelle where there is sufficient concentration of water but with increased activity than in the bulk water phase. As in CTAB and non-micellar media, the reaction is also free-energy controlled in SDS thereby suggesting the same mechanism in the three media. Similarly, $\Delta H^*$ values are lower while $\Delta S^*$ values are higher in SDS than in non-micellar medium (Table 4).
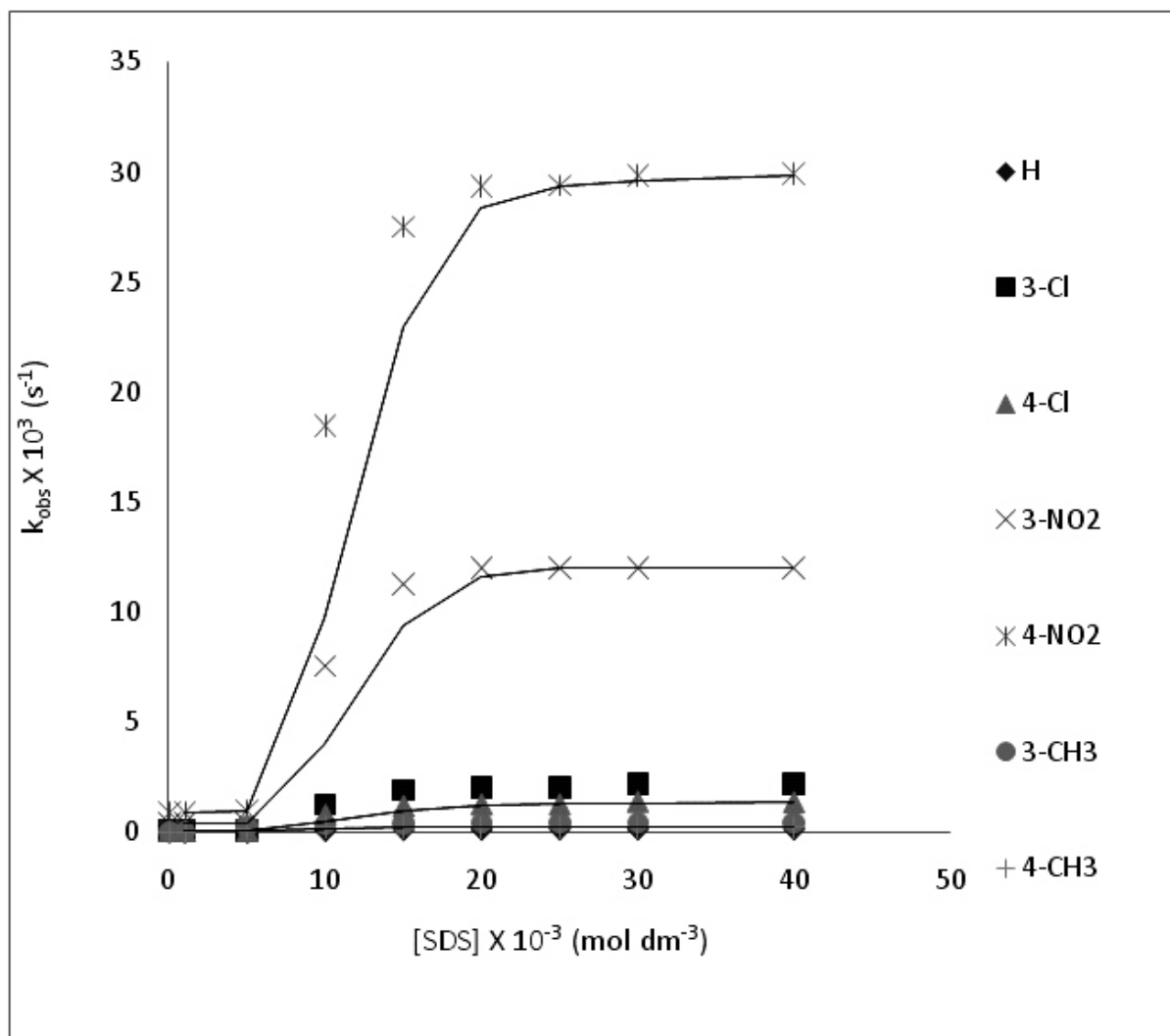
**Figure 5: Variation of $k_{obs}$ with [SDS] at Fixed Substituted Phenyl Trifluoroacetate Concentration (5.0 X $10^{-4}$ mol dm$^{-3}$) at 25$^o$C**

**Table 4: Activation Parameters (ΔH* and ΔS*) in the Hydrolysis of Fixed Substituted Phenyl Trifluoroacetate Concentration (5.0 x$10^{-4}$ moldm$^{-3}$) and Fixed SDS concentration (1.0 x $10^{-3}$ mol. dm$^{-3}$)**

| Phenyl substituent X | $k_{obs}$ x$10^4$(s$^{-1}$)at 25$^o$C | ΔH* ( kJmol$^{-1}$) | ΔS* (Jmol$^{-1}$K$^{-1}$) | log $k_{obs}$ | $\sigma_x$ |
|---|---|---|---|---|---|
| H | 1.58 ± 0.01 | +24.7± 0.2 | -205 ± 2 | -3.80 | 0.00 |
| 3-Cl | 11.22 ±0.02 | +25.9 ± 0.3 | -189 ± 2 | -2.95 | 0.37 |
| 4-Cl | 5.36 ± 0.01 | +27.4 ± 0.2 | -204 ± 2 | -3.27 | 0.23 |
| 3-NO$_2$ | 68.08 ±0.02 | +27.4± 0.2 | -185 ± 1 | -2.61 | 0.71 |
| 4-NO$_2$ | 97.72 ±0.02 | +31.8± 0.2 | -172 ± 2 | -2.01 | 0.78 |
| 3-CH$_3$ | 1.09 ±0.02 | +20.2 ± 0.1 | -225 ± 1 | -3.96 | -0.07 |
| 4-CH$_3$ | 0.64 ±0.02 | +22.7± 04 | -219 ± 2 | -4.19 | -0.17 |

The entropy increase is even greater in SDS than in CTAB vis-à-vis non-micellar medium, suggesting that the solvation of the transition state with respect to the reactants is smaller in SDS than in CTAB. This also implies that the reactions are faster in SDS medium than in non-micellar medium. Figure 6 shows the Hammett plot for the reactions in SDS.
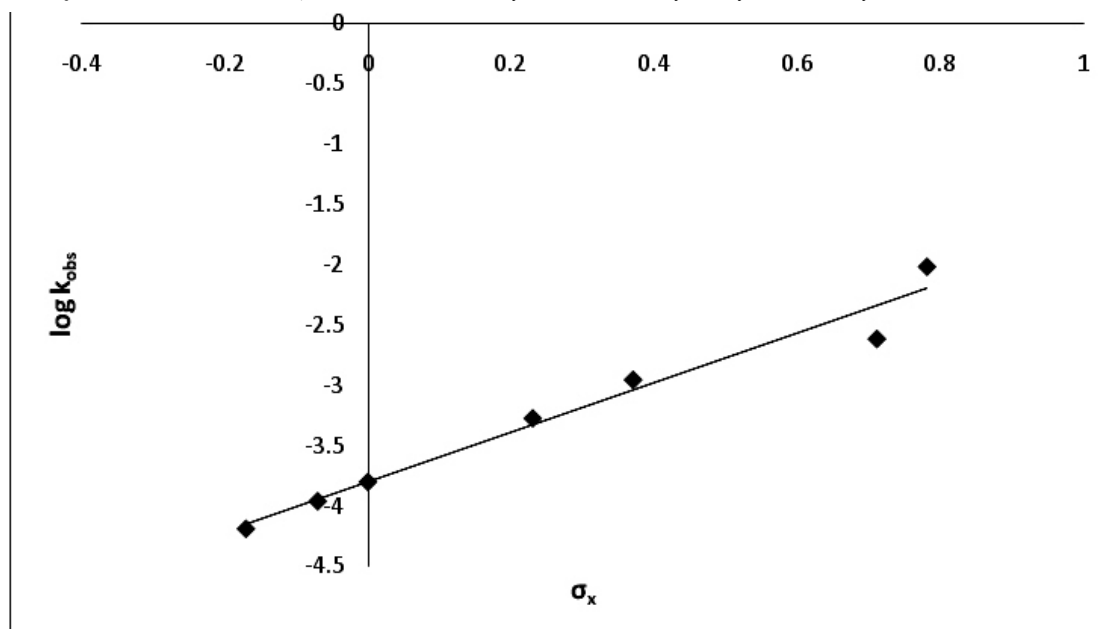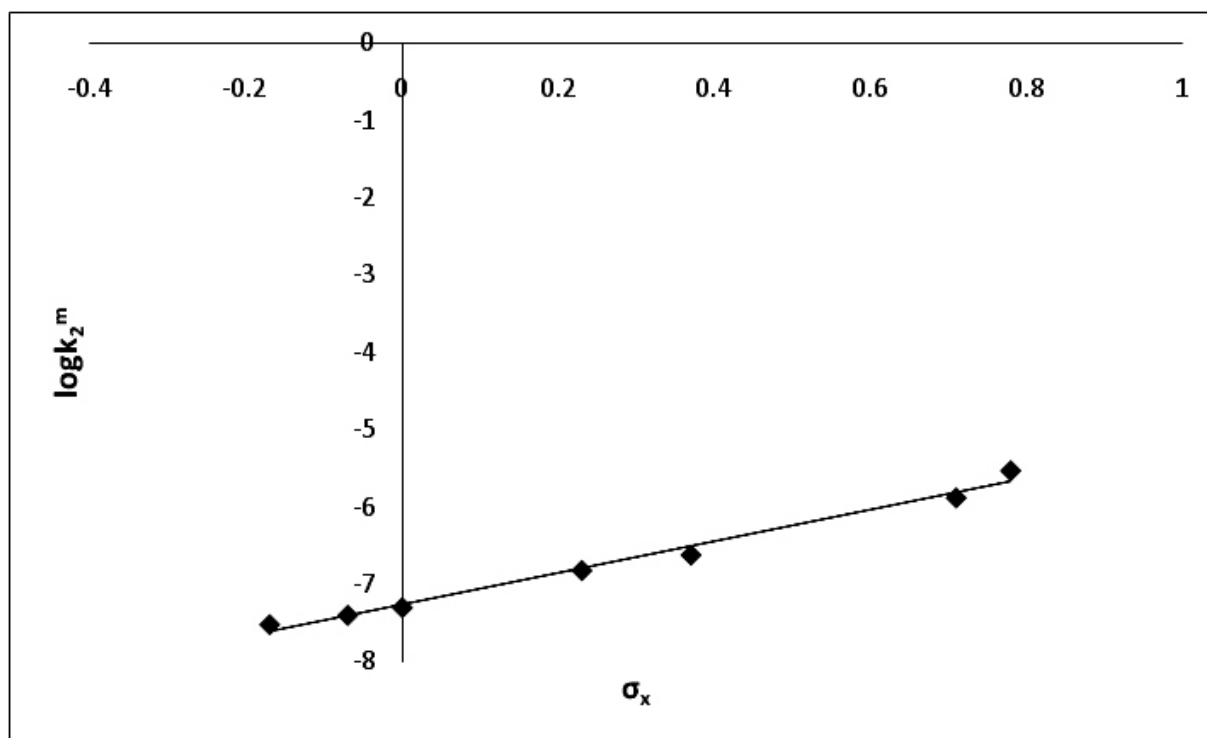
**Figure 6: Hammett-Type log $k_{obs}$ vs Substituent Constants ($\sigma_x$) at Fixed Concentration ($5.0 \times 10^{-4}$ mol dm$^{-3}$) of Substituted Phenyl Trifluoroacetates and Fixed SDS concentration ($3.0 \times 10^{-3}$ mol dm$^{-3}$) at 25°C.**

The reaction constant (r) here is 2.30. This shows that the reaction also responds to substituent effects in SDS in a way similar to that in CTAB and aqueous media. The compliance with Hammett relationship here suggests that the same mechanism operates in SDS, CTAB and non-micellar media. However, the value of r in SDS implies that the hydrolysis of the esters is less susceptible to substituent effects in SDS than in CTAB or non-micellar medium. This further supports the fact that the reactions are faster in CTAB micelles than in SDS or non-micellar medium. The observation in fact is in line with the Hammond Postulate (Hammond, 1955).

As in CTAB, the second order rate constants were calculated (Table 5). A plot of log $k_2^m$ versus Hammett substituent constants ($\sigma_x$) in SDS is presented in Figure 7. From the plot, a reaction constant of +2.18 is obtained. This smaller value of here indicates that the reaction's susceptibility to substituent effects is less in the micelle pseudophase than in gross micellar medium. That a linear relationship is also obtained here with positive reaction constant indicates that the reaction occurs in the pseudophases as in the entire SDS micellar medium.

**Table 5: Second Order Rate Constants ($k_w$, $k_M$, $k_2^m$) at Fixed SDS Concentration ($1.0 \times 10^{-2}$ mol dm$^{-3}$) and Fixed concentration ($5.0 \times 10^{-4}$ mol dm$^{-3}$) of Substituted phenyl Trifluoroacetates at 25°C.**

| Phenyl substituent X | $k_w$ ($\times 10^6$) mol$^{-1}$dm$^3$s$^{-1}$ | $k_m$ ($\times 10^6$) mol$^{-1}$dm$^3$s$^{-1}$ | $k_2^m$($\times 10^7$) mol$^{-1}$ dm$^3$s$^{-1}$ | log$k_2^m$ | $\sigma_x$ |
|---|---|---|---|---|---|
| H | 0.25 | 0.4 | 0.5 | -7.30 | 0.00 |
| 3-Cl | 2.20 | 1.7 | 2.4 | -6.62 | 0.37 |
| 4-Cl | 1.37 | 1.1 | 1.5 | -6.82 | 0.23 |
| 3-NO$_2$ | 14.65 | 9.5 | 13.3 | -5.88 | 0.71 |
| 4-NO$_2$ | 30.06 | 20.9 | 29.2 | -5.53 | 0.78 |
| 3-CH$_3$ | 0.18 | 0.3 | 0.4 | -7.40 | -0.07 |
| 4-CH$_3$ | 0.11 | 0.2 | 0.3 | -7.52 | -0.17 |

**Figure 7 : Hammett-type log $k_2^m$ vs Substituent Constants ($\sigma_x$) at Fixed Concentration (5.0 X $10^{-4}$ mol dm$^{-3}$) of Substituted Phenyl Trifluoroacetates and Fixed SDS Concentration (1.0 X $10^{-3}$ mol dm$^{-3}$) at 25°C.**

## CONCLUSION

In this work, we have shown that the neutral hydrolysis of aryl trifluoroacetates is catalyzed by micelles of CTAB and SDS. The reaction mechanism is the same in the two micellar media, and indeed in the micellar pseudophases of these micelles, as in aqueous medium. As in aqueous medium, the reactions obey the Hammett relationship in the micellar pseudophase, demonstrating the applicability of this important Linear Free Energy relationship to reactions occurring in micelles.

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