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Research Paper

Investigation of Acid-Catalyzed Hydrolysis of 4-Nitrophenyl-*N*-Acetyl-4-Methylbenzenesulfonimidate in Dioxane-Water Solutions

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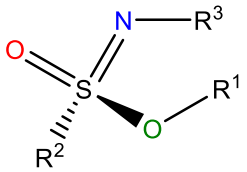
Abstract: In this study, the change of the hydrolysis mechanism of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate was investigated in different solvent ratios. For this, the mechanism was investigated in 20%, 40%, and 60% (v/v) dioxane-water solutions of mineral acids. 20% and 40% (v/v) dioxane over a wider acidity range and rate maxima were observed for perchloric and sulfuric acids. Similarly, rate maxima are observed perchloric and hydrochloric acid in 60% (v/v) dioxane at high acid concentration. Analyses of the data by the Cox–Yates excess acidity method and temperature effects indicate hydrolysis by an A-2 mechanism. It was determined that the mechanism did not change when different solvent ratios were used.

Keywords: Sulfonimidate, Acid-catalyzed, Hydrolysis, A-2 mechanism, Excess Acidity

1. Introduction

In the literature, the mechanism of acid-catalyzed hydrolysis has been studied in aqueous solutions. In this study, it is aimed to determine the acid-catalyzed hydrolysis mechanism in different solvent mixtures. Thus, it will be possible to determine the acid-catalyzed hydrolysis mechanism for substances that are completely insoluble in pure water.

Sulfonimidates, which are mono-aza analogs of sulfonamides, are useful reagents for organic synthesis [1]. Sulfonimidates, a tetrahedral sulfur(VI) centered are compounds to which four different groups (as shown in Scheme 1) are attached [2]. With the discovery of sulfonimidate synthesis 50 years ago, allowed intensive study of this class of organo sulfur compounds [3].



R^1, R^2 = Alkyl, Aryl

R^3 = H (NH-sulfonimidate)
or R^3 = Aryl, Acyl, Alkyl,
Cyano, Sulfonyl, Halo,
Amino, Phosphonyl

Scheme 1. General overview of the sulfonimidate structure.

Sulfonimidates are a class of chiral molecules with very interesting structures that have applications in materials science. Polyorganooxzothiazines obtained from thermally induced condensation of sulfonimidates have been studied as thermally stable and solvent resistant sulfur(VI)-nitrogen backbone polymers [4]. Sulfonimidates were obtained from sulfinamides and iodosobenzene in high yields in a single step under mild conditions, with this reaction, it provides fast and efficient access

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to a class of molecules of important synthetic, biological and industrial importance [5]. These compounds are also known to exhibit human carbonic anhydrase II inhibitory activity [6].

Acid-catalyzed reactions occur in two types, general and specific acid catalysis. In general acid catalysis, the catalysis proceeds according to the properties of the acidic species. A reaction in which reaction rate depends not only upon the concentration of hydrogen ion but also on the concentration of all acids present in the solution is said to follow general acid catalysis. In specific acid catalysis, the rate-determining step is the protonation of the substrate. The substrate is rapidly protonated. The protonated substrate is converted to the product in a slow step. Depending on whether the rate-determining step is monomolecular or bimolecular, the reaction follows an A-1 or A-2 mechanism [7-8].

(4-Methoxybenzoyl)-4-toluenimino sulfonate [9], amidosulfites [10], Substitutedarylthio phthalimides [11], and *N*-(4-Substitutedaryl) succinimides [12] kinetic studies have been observed in the literature.

Our interest in determining the mechanism for completely water-insoluble compounds led us to this study. In this study, the hydrolysis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in 20%, 40%, and 60% (v/v) dioxane-water solutions of mineral acids was investigated.

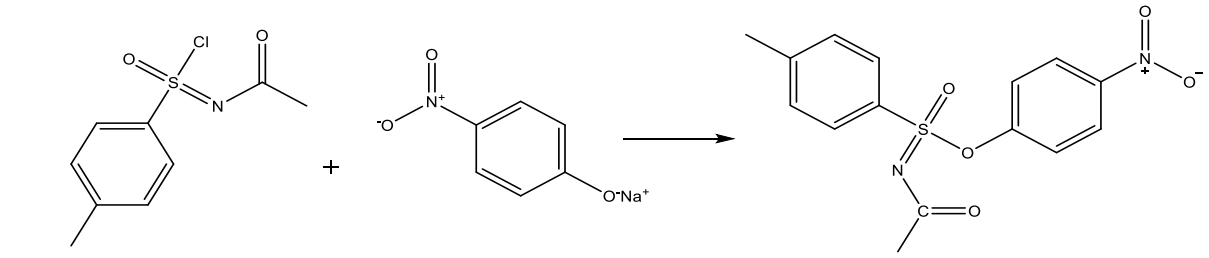
2. Experimental Methods

2.1. Materials

All chemicals were used without purification since they were supplied in high purity. The physical data of the synthesized products were compared with the samples prepared by classical methods. The melting point was determined with the SMP30 Stuart Digital Melting Point instrument. All kinetic measurements of the 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate were made with the Thermo Evolution 220 Ultraviolet-Visible Spectrophotometer.

2.2. Synthesis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate

4-Nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate was synthesized according to the literature [13].



N-Acetyl-*p*-toluenesulfonimidoyl chloride *p*-Nitrophenoxide sodium salt 4-Nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate

Scheme 2. Synthesis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate

A benzene (Merck, 99.9%) solution of *N*-acetyl-*p*-toluenesulfonimidoyl chloride (0.01 mol) was added to a suspension of *p*-nitrophenoxide sodium salt (Sigma-Aldrich, 95%) (0.01 mol) in 15 mL benzene (Merck, 99.9%). The reaction mixture was heated for 90 minutes. The form sodium chloride was filtered off with suction method. The filtrate was evaporated on a water bath in a rotary evaporator. The residual oil was triturated with petroleum ether (60-80, ligroin) (Sigma-Aldrich,

puriss) to produce crystals. Recrystallization from methanol (Merck, 99.9%) gave the ester, yield 2.2g (66%), mp. 87 °C. (Scheme 2)

2.3. Kinetic Studies

A stock solution of ester in 1,4-dioxane (Merck, 99.9%) was prepared ($1x10^{-2}$M) and used within a week. The reaction solution of mineral acid (5.00 M) containing 40%(v/v) dioxane-water was pipetted (3.00 mL) into a 1.0 cm silica cell which was positioned in the cell compartment and allowed to reach the reaction temperature (25.0±0.1°C) over 15 minutes. Without removal, 30µL of stock solution were injected into the reaction solution using a 50 µL Hamilton syringe, mixed using a teflon stirrer and copped. The rate of reaction was followed on a Thermo Evolution 220 UV-Vis. Spectrophotometer; the change in the optical density was recorded as a function of time.

3. Results and Discussion

3.1.Calculation of Rate-Coefficients

Pseudo first-order rate-coefficients were determined from plots of $In(A_\infty - A_t)$ against *time*, where A_t is the optical density at time *t*, and A_∞ is the optical density at infinity (Eq.1). For a pseudo first-order reaction:

$$rate = \frac{-d[A]}{[A_0]} = k_1.dt$$

$$\int_{A_0}^{A} \frac{d[A]}{[A_0]} = \int_{t}^{\infty} -k_1 dt$$

$$ln\ [A] - ln\ [A_0] = -k_1.t$$

$$ln\,[A] = ln\ [A_0] - k_1.t$$

$$ln\,[A] = -k_1\,.t + constant \qquad (1)$$

A plot of *lnA* versus *time* will give a straight line of slope $-k_1$. The infinity reading of A_∞ was taken after at least ten half-lives.

Table 1. Effect of concentration of various mineral acids on the acid catalysed hydrolyses of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in the presence of 20% dioxane-water (v/v) and at 25.0±0.1°C

[M]	[HCl], 10^3k_1(sec^{-1})	[H_2SO_4], 10^3k_1(sec^{-1})	[$HClO_4$], 10^3k_1(sec^{-1})
1.00	1.42	1.53	1.49
1.50	2.40	2.57	2.26
2.00	3.64	3.77	3.42
2.50	4.87	5.27	4.29
3.00	6.64	7.47	5.36
3.50	7.94	8.00	6.11
4.00	9.04	8.86	7.14
5.00	13.4	10.9	8.17
6.00	17.3	12.0	7.67
7.00	18.0	8.85	3.87
8.00	18.3	5.62	1.65

Effect of concentration of various mineral acids on the acid catalysed hydrolyses of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in the presence of 20%, 40%, and 60% dioxane-water (v/v) and at 25.0±0.1°C is shown Table 1, 3, and 5, respectively. Values of $10^3k_1(sec^{-1})$ for the acid catalysed hydrolyses of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in 20%, 40%, and 60% dioxane-water (v/v) and at different temperatures is shown Table 2, 4, and 6, respectively.

Table 2. Values of $10^3k_1(sec^{-1})$ for the acid catalysed hydrolyses of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in 20% dioxane-water (v/v) and at different temperatures.

Acid	$[H^+]$ / M	20.0 °C	25.0 °C	30.0 °C	35.0 °C	40.0 °C
HCl	2.00	2.37	3.64	5.56	8.06	11.4
	3.00	4.24	6.64	9.81	14.5	21.0
	4.00	5.87	9.04	14.0	20.4	29.0
H_2SO_4	2.00	2.42	3.77	5.70	8.23	11.6
	3.00	4.79	7.47	10.9	15.9	23.2
	4.00	5.66	8.86	13.0	19.3	28.4
$HClO_4$	2.00	2.26	3.42	5.13	7.48	103
	3.00	3.39	5.36	7.83	11.8	17.6
	4.00	4.36	7.14	10.8	16.4	24.8

Table 3. Effect of concentration of various mineral acids on the acid catalysed hydrolyses of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in the presence of 40% dioxane-water (v/v) and at 25.0±0.1°C

[M]	[HCl], $10^3k_1(sec^{-1})$	[H_2SO_4], $10^3k_1(sec^{-1})$	[$HClO_4$], $10^3k_1(sec^{-1})$
1.00	1.22	1.27	1.56
1.50	2.03	2.24	2.48
2.00	3.18	3.48	3.75
2.50	4.80	5.23	5.00
3.00	6.32	6.85	6.27
3.50	7.88	7.73	7.38
4.00	10.5	9.30	8.93
5.00	13.5	11.5	8.12
6.00	-	8.35	4.72
7.00	16.4	1.62	2.04
8.00	-	-	0.83
9.00	-	-	0.32

Table 4. Values of $10^3k_1(sec^{-1})$ for the acid catalysed hydrolyses of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in 40% dioxane-water (v/v) and at different temperatures.

Acid	$[H^+]$ / M	20.0 °C	25.0 °C	30.0 °C	40.0 °C
HCl	2.00	2.14	3.18	4.71	10.2
	3.00	4.29	6.32	9.51	20.3
H_2SO_4	2.00	2.30	3.48	5.13	11.4
	3.00	4.64	6.85	10.2	22.0
$HClO_4$	2.00	2.40	3.75	5.75	12.5
	3.00	4.17	6.27	9.61	21.9

Table 5. Effect of concentration of various mineral acids on the acid catalysed hydrolyses of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in the presence of 60% dioxane-water (v/v) and at 25.0±0.1°C

[M]	[HCl], 10^3k_1(sec^{-1})	[H_2SO_4], 10^3k_1(sec^{-1})	[$HClO_4$], 10^3k_1(sec^{-1})
0.60	0.43	0.44	0.69
0.80	-	0.69	-
1.00	0.83	0.94	1.49
1.20	-	-	2.18
1.30	-	1.33	-
1.40	-	-	2.56
1.50	1.68	1.88	-
1.60	-	-	3.18
1.70	-	2.33	-
1.85	-	-	4.02
2.00	2.52	3.11	4.51
2.20	-	-	5.34
2.40	-	-	6.05
2.50	4.21	5.22	6.31
2.60	-	-	6.78
2.80	-	-	7.73
2.90	-	-	8.83
3.00	5.94	7.87	10.3
3.50	-	-	10.2
4.00	11.3	10.6	8.82
4.25	12.9	-	-
4.50	12.7	-	6.91
4.75	11.7	-	-
5.00	8.16	13.1	5.42

Table 6. Values of 10^3k_1(sec^{-1}) for the acid catalysed hydrolyses of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in 60% dioxane-water (v/v) and at different temperatures.

Acid	[H^+] / M	20.0 °C	25.0 °C	30.0 °C	35.0 °C	40.0 °C
HCl	1.00	-	0.83	1.23	2.75	4.00
	2.00	1.69	2.52	3.78	8.34	-
	2.50	2.69	4.21	6.14	13.7	-
H_2SO_4	1.00	-	0.94	1.44	3.19	4.68
	2.00	1.99	3.11	4.62	10.2	-
	2.50	3.34	5.22	7.91	18.0	-
$HClO_4$	1.00	-	1.49	2.26	5.14	7.54
	2.00	2.96	4.51	6.64	1.51	-
	2.50	4.05	6.31	9.51	21.4	-

The hydrolysis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in 20%, 40%, and 60% (v/v) dioxane-water solutions of mineral acids are shown in Figures 1, 2, and 3, respectively. The kinetic results are similar to those reported for the hydrolysis of *p*-nitrophenyl *N*-acetyl-phenyl iminosulfonate in 20% (v/v) dioxane [14]. Maximum rates were observed for perchloric and sulfuric acids, as hydrolysis was studied on a broader acidity range at 20%, and 40% (v/v) dioxane. Similarly rate maxima are observed perchloric and hydrochloric acid in 60% (v/v) dioxane at high acid concentration. Hydrolyses of amides [15], and *N*-sulfonyl sulfilimines [16] was observed that a_{H2O} value decreased with increasing acid concentration.

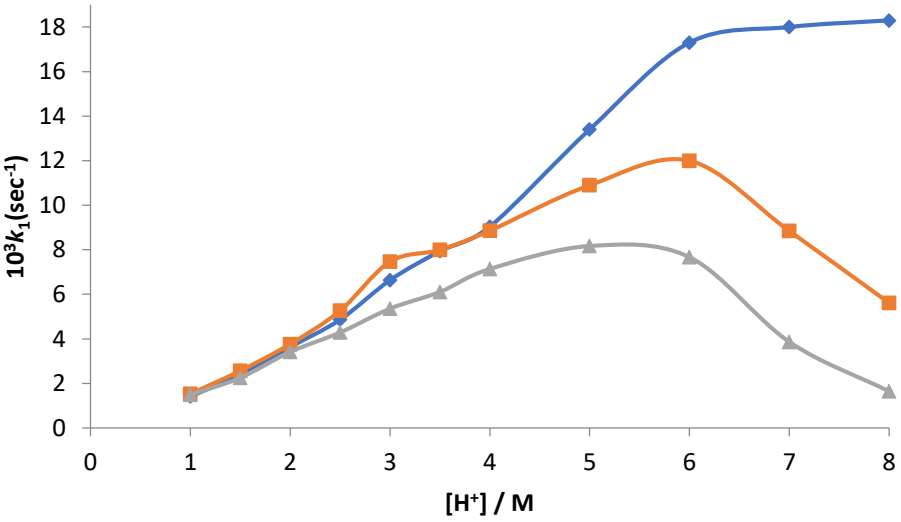


Figure 1. The acid-catalysed hydrolysis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in 20% (v/v) dioxane (data from Table 1) ♦ HCl , ■ H_2SO_4, ▲ $HClO_4$

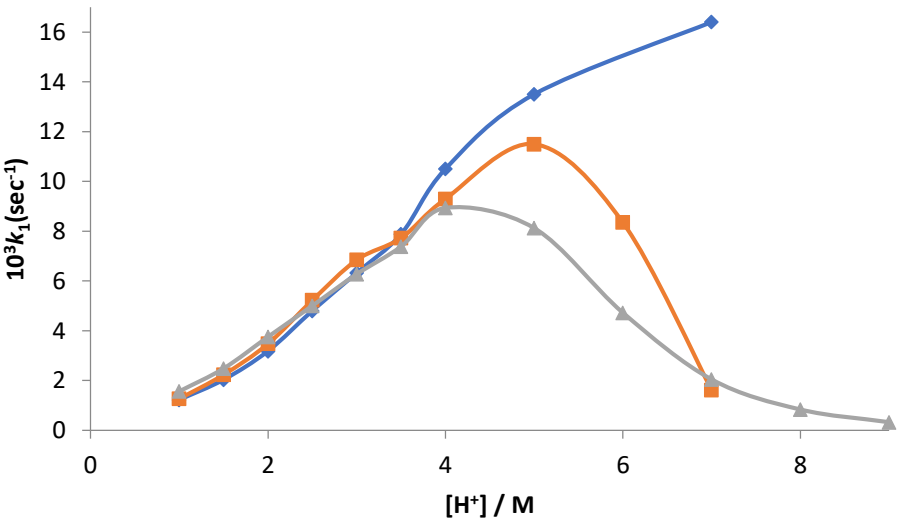


Figure 2. The acid-catalysed hydrolysis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in 40% (v/v) dioxane (data from Table 3) ♦ HCl , ■ H_2SO_4, ▲ $HClO_4$

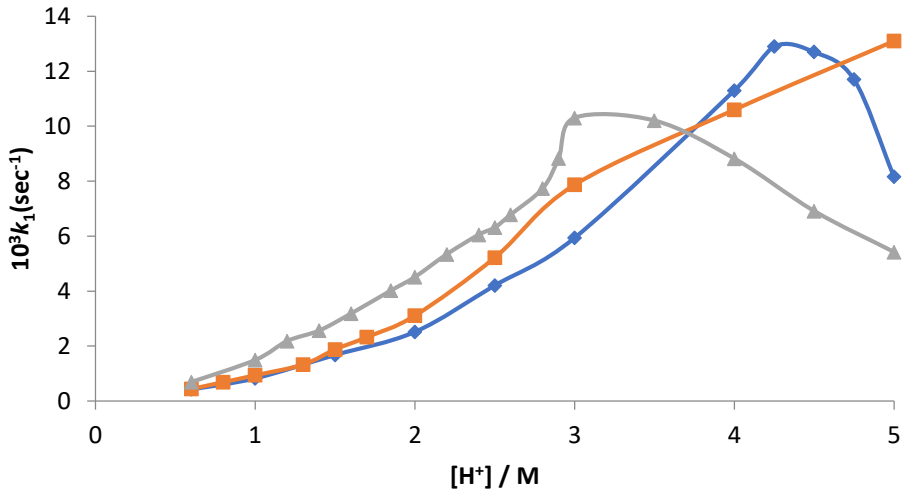


Figure 3. The acid-catalysed hydrolysis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in 60% (v/v) dioxane (data from Table 5) ♦ HCl , ■ H_2SO_4, ▲ $HClO_4$

3.2. Entropies of Activation

In the study, the relationship between rate constants and temperature was investigated with Eyring equation. Arrhenius parameter values at different temperatures are shown in Tables 7, 8, and 9, respectively.

Table 7. Arrhenius parameters for the hydrolysis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in 20% dioxane-water (v/v), at 25.0°C (Data from Table 2).

Acid	[H$^+$] / M	$\Delta H^{\neq}$(kJmol^{-1})	$\Delta S^{\neq}$(JK^{-1}mol^{-1})
HCl	2.00	57.68 ± 0.96	-97.94 ± 3.18
	3.00	58.23 ± 0.63	-91.12 ± 2.10
	4.00	58.73 ± 1.04	-86.82 ± 3.51
H_2SO_4	2.00	55.68 ± 1.08	-105.10 ± 3.64
	3.00	59.82 ± 0.75	-87.65 ± 2.55
	4.00	63.28 ± 0.84	-73.65 ± 2.76
$HClO_4$	2.00	57.18 ± 1.21	-99.32 ± 4.01
	3.00	57.14 ± 0.75	-93.97 ± 2.47
	4.00	58.60 ± 0.63	-87.53 ± 2.00

Table 8. Arrhenius parameters for the hydrolysis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in 40% dioxane-water (v/v), at 25.0°C (Data from Table 4).

Acid	[H$^+$] / M	$\Delta H^{\neq}$(kJmol^{-1})	$\Delta S^{\neq}$(JK^{-1}mol^{-1})
HCl	2.00	57.18 ± 0.58	-100.60 ± 1.96
	3.00	56.97 ± 0.58	-95.55 ± 1.92
H_2SO_4	2.00	60.27 ± 0.71	-88.95 ± 2.38
	3.00	61.07 ± 0.92	-81.89 ± 3.05
$HClO_4$	2.00	58.56 ± 0.75	-95.35 ± 2.47
	3.00	57.02 ± 0.67	-94.72 ± 2.17

Table 9. Arrhenius parameters for the hydrolysis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in 60% dioxane-water (v/v), at 25.0°C (Data from Table 6).

Acid	$[H^+]$ / M	$\Delta H^{\neq}$ (kJmol^{-1})	$\Delta S^{\neq}$ (JK^{-1}mol^{-1})
HCl	1.00	59.77 ± 0.54	-103.16 ± 1.76
	2.00	58.56 ± 0.71	-97.94 ± 2.34
	2.50	59.19 ± 0.88	-91.80 ± 2.97
H_2SO_4	1.00	61.61 ± 0.29	-92.20 ± 0.92
	2.00	59.52 ± 1.00	-89.91 ± 3.30
	2.50	60.90 ± 0.33	-82.51 ± 1.04
$HClO_4$	1.00	60.69 ± 0.17	-99.10 ± 0.54
	2.00	59.48 ± 0.54	-93.17 ± 1.84
	2.50	61.73 ± 0.33	-81.34 ± 1.09

Activation entropy values obtained in the study of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate were $\Delta S^{\neq}$ = -97.94, -105.10, and -99.32 JK^{-1}mol^{-1} for 2.00 M hydrochloric, perchloric and sulfuric acids, respectively, in 20% dioxane (v/v). Similarly, the activation entropy values obtained in 40% dioxane (v/v) were $\Delta S^{\neq}$ = -100.60, -88.95, and -95.35 JK^{-1}mol^{-1} for 2.00 M hydrochloric, perchloric and sulfuric acids, respectively. In 60% dioxane (v/v) values of entropy of activation for hydrolysis of the esters $\Delta S^{\neq}$ = -97.94, -89.91, and -93.17 JK^{-1}mol^{-1} for 2.00 M hydrochloric, perchloric and sulfuric acids, respectively (Tables 7, 8, and 9) are also consistent with a bimolecular mechanism.

3.3. Excess Acidity Method

Kinetic data in Table 10 was analysed by the Excess Acidity treatment of Cox and Yates [17]. The appropriate kinetic equation for mainly unprotonated substrates Eq. 2 was used.

$$\log k_1 - \log C_{H^+} - [\log Cs / (Cs + C_{SH^+})] = m^* m^{\neq} X + r \log a_{Nu} + \log (k_o / K_{SH^+}) \quad (2)$$

Table 10. The acid-catalysed hydrolysis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in perchloric acid, 60% (v/v) dioxane at 25.0°C

$[H^+]$ / M	log$[H^+]$	logk_1	logk_1-log$[H^+]$	X
1.00	0.000	-2.827	-2.287	0.200
1.40	0.146	-2.592	-2.738	0.313
1.60	0.204	-2.496	-2.700	0.360
1.85	0.267	-2.396	-2.663	0.447
2.00	0.301	-2.346	-2.647	0.493
2.20	0.342	-2.272	-2.614	0.557
2.40	0.380	-2.218	-2.598	0.630
2.50	0.398	-2.200	-2.598	0.667

Due to the extremely low basicity of the sulfonimidic ester studied, the protonation correction term could be neglected. Values of Excess acidity (*X*) for aqueous solutions of acid were used [18]. A plot of logk_1-log[H^+] against *X* is shown in Figure 4 for the hydrolysis of the ester in perchloric acid.

The resulting curved plot is typical of an A-2 reaction involving water in the rate-determining transition state.

As seen in Figure 4, the rate of hydrolysis increases with the increase in acid concentration. A curve in this figure shows that the reaction proceeds by the A-2 mechanism. According to Figure 5, when 2 moles of water activity is subtracted, a straight line is obtained. Thus, it is understood that 2 moles of water are involved in the rate-determining step via an A-2 mechanism.

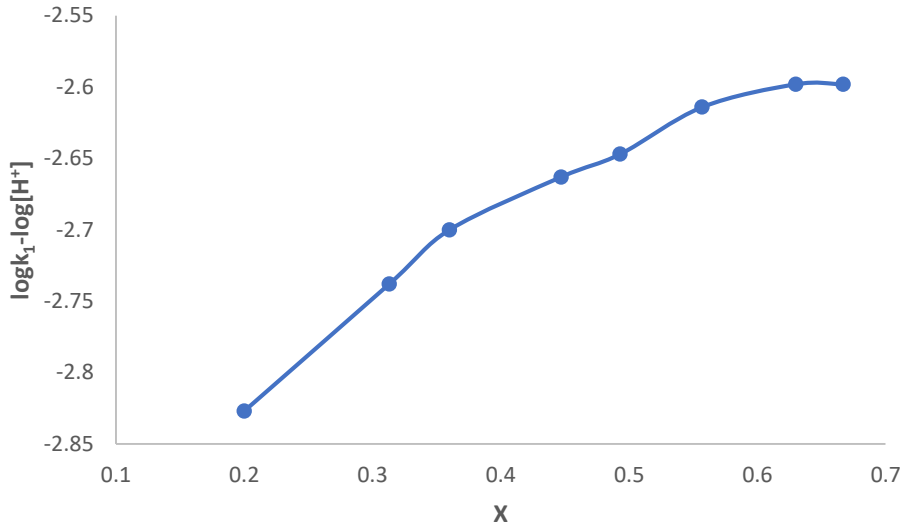


Figure 4. Plot of logk_1-log[H^+] versus X for the hydrolysis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in perchloric acid (Data from Table 10)

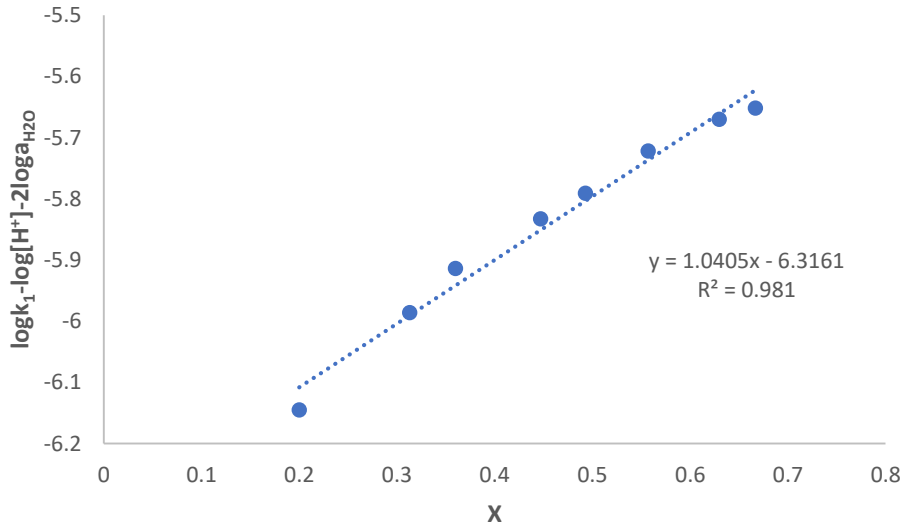
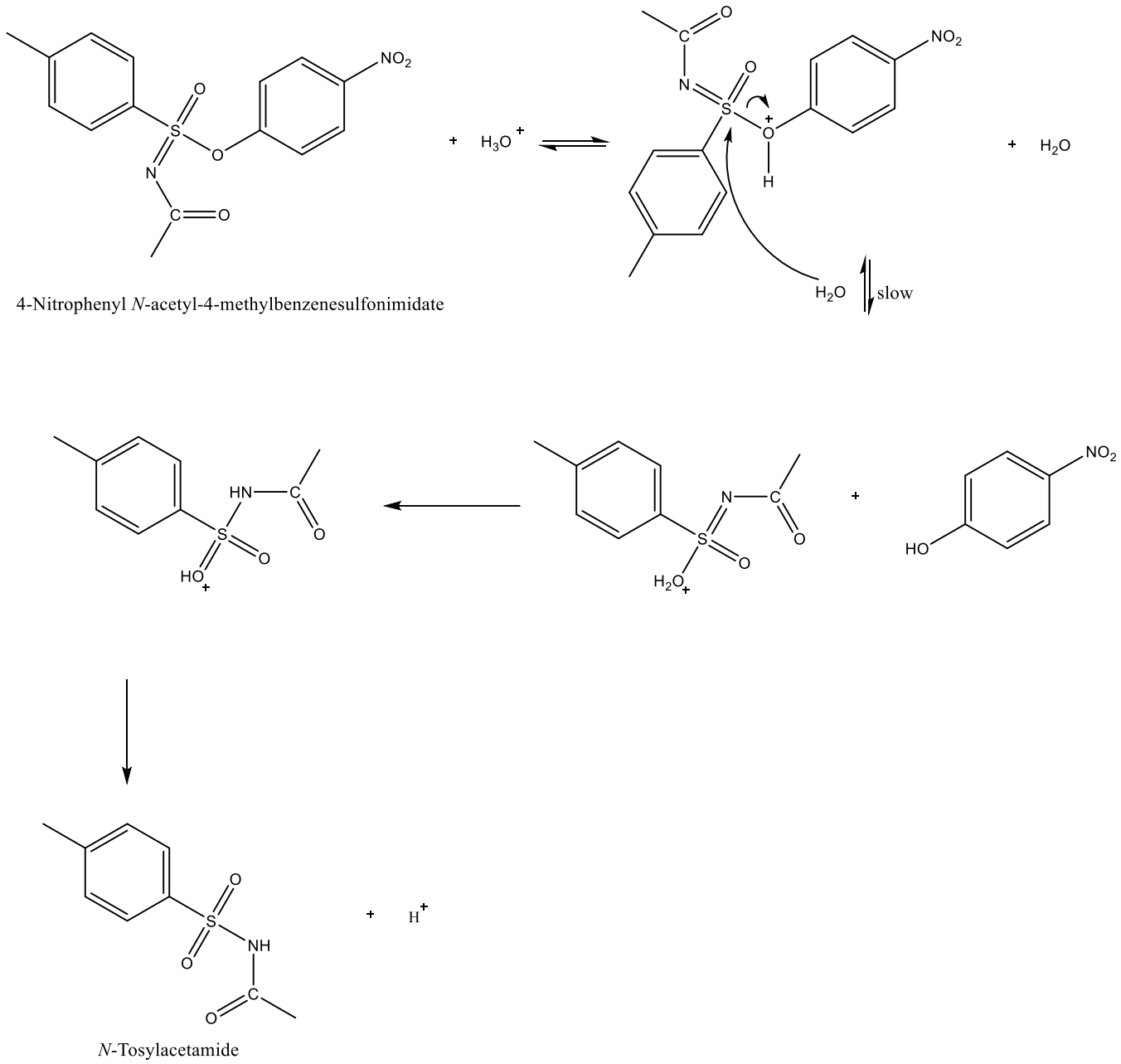


Figure 5. Plot of logk_1-log[H^+]-2loga_{H2O} versus X for the hydrolysis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate in perchloric acid.

These data confirm that the hydrolysis mechanisms of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate are similar in 20%, 40%, and 60% dioxane (v/v). Although there is no

direct evidence for site of protonation on *N*-acetylsulfonimidic ester, a possible mechanism for hydrolyses of the ester involving protonation on oxygen is shown in Scheme 3.



Scheme 3. A plausible acid-catalyzed hydrolysis mechanism of the 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate.

3. Conclusions

The acid-catalyzed hydrolysis of 4-nitrophenyl *N*-acetyl-4-methylbenzenesulfonimidate was studied for the first time. Analysis of the data obtained as a result of the examination in 20%, 40% and 60% (v/v) dioxane-water acid solutions with the Cox-Yates excess acidity method and temperature effects indicate hydrolysis with an A-2 mechanism. In this study, it has been seen that the mechanism determination can be made for water-insoluble compounds using different binary solvent systems. Thus, by directing different studies, it is possible to try to determine the mechanism by using alcohol-water or acetonitrile-water solvent systems. The reaction mechanism was determined by kinetic studies and brought to the literature.

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Authors' Contributions

SÖ and HK designed the experimental stages. SÖ did the experimental work, kinetic calculations and wrote the article. Both authors read and approved the final draft.

Competing Interests

The authors declare that they have no competing interests.

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