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Halil Kutuk^a & Hasan Yakan^a

^a Ondokuz Mayıs University, Faculty of Arts and Sciences, Chemistry Department, Kurupelit, Samsun, Turkey

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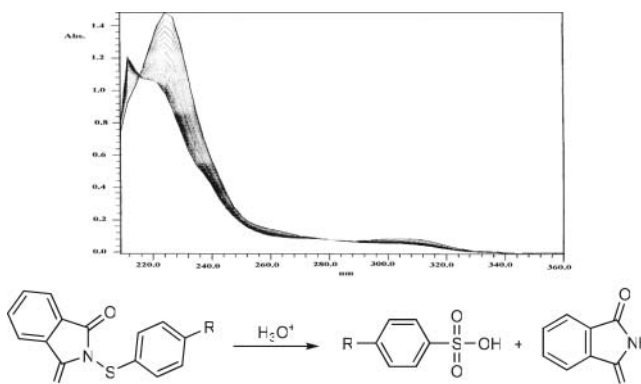
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THE MECHANISMS OF ACID-CATALYZED HYDROLYSIS OF N-(4-SUBSTITUTED ARYLTHIO) PHTHALIMIDES

Halil Kutuk and Hasan Yakan

Ondokuz Mayıs University, Faculty of Arts and Sciences, Chemistry Department,
 Kurupelit, Samsun, Turkey

GRAPHICAL ABSTRACT



Abstract The acid-catalyzed hydrolysis of N-(4-substituted arylthio)phthalimides was studied in aqueous solutions of sulfuric, perchloric, and hydrochloric acids at $40.0 \pm 0.1^\circ\text{C}$. Analysis of the data by the excess acidity method, activation parameters, and substituent effects indicates hydrolysis by an A-2 mechanism at low acidity. At higher acidities, a changeover to an A-1 mechanism is observed.

Keywords Acid-catalyzed hydrolysis; arylthiophthalimides; excess acidity; sulfenimides

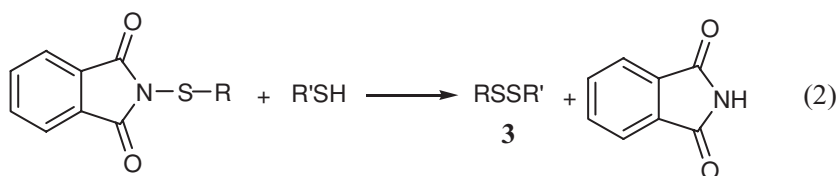
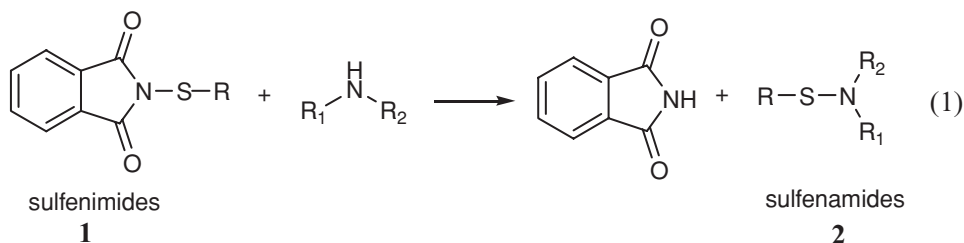
INTRODUCTION

N- of trichloromethanesulfonylchloride with sodium phthalimide was the first sulfenimide(thiophthalimide) **1** reported in the literature. The synthesis of some arylthiophthalimides was reported by Klivenye and coworkers.² Several sulfenimides were prepared by Büchel and Conte³ by the reaction of disulfides with the desired N-bromoimides. Alkyl and aryl sulfenimides have been synthesized by the reaction of sulfonyl chlorides with imides in the presence of a tertiary amine.⁴ The stability of sulfonyl chloride solutions has been found to be dependent on the temperature and the polarity of the solvent.⁴ Some sulfenimides

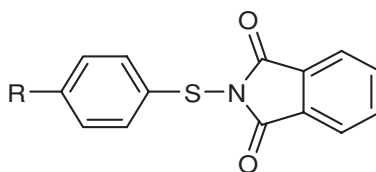
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Address correspondence to Prof. Dr. Halil Kutuk, OMU Fen-Ed. Fakültesi, Kimya Bölümü, Kurupelit, Samsun 55139, Turkey. E-mail: hkutuk@omu.edu.tr

function as sulfenyl transfer reagents and can be used to prepare sulfenamides **2**. When thiophthalimides react with thiols to form unsymmetrical disulfides **3** and phthalimide. The thiophthalimides are the best known of these reagents (Eqs. (1) and (2)).^{5,6}



A study of the kinetics and mechanism of the acid-catalyzed hydrolysis of a series of N-(4-substituted arylsulfinyl)phthalimides,⁷ amidosulfites,⁸ N-(4-substituted arylsulfonyl)phthalimides,⁹ arenesulfenamides,¹⁰ and benzohydroxamic acids¹¹ have been studied in concentrated aqueous mineral acids in our laboratory. There has been no systematic study of the acid-catalyzed hydrolysis of N-(4-substituted arylthio)phthalimides in highly acidic solutions. We report a complementary study of acid-catalyzed hydrolysis of a series of N-(4-substituted arylthio)phthalimides (**4a-c**) in concentrated aqueous mineral acids as shown in Scheme 1.



- 4 a:** R = H
b: R = CH₃
c: R = Cl

Scheme 1

RESULTS AND DISCUSSION

The first-order rate coefficients, k_1 , for the hydrolysis of N-(4-methylphenylthio)phthalimide **4b** in aqueous solutions of mineral acids are shown in Figure 1. Rate maxima are observed for both perchloric and hydrochloric acid because hydrolysis was studied

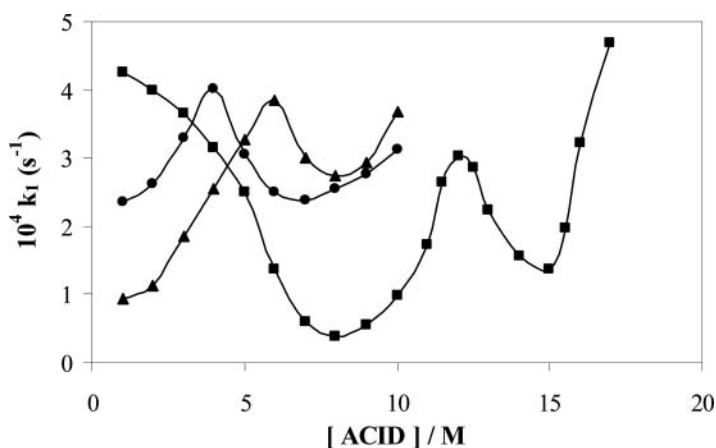


Figure 1 Plots of k_1 for the acid-catalyzed hydrolysis of **4b** in aqueous acid solutions at 40.0 °C; ●, HCl; ■, H₂SO₄; ▲, HClO₄.

over a wider range of acidity. In all cases, the rates of hydrolysis decrease continuously with increasing concentration of sulfuric acid up to 7.00 M, whereas at acid concentrations above 8.00 M the rate increases and there is an indication of a rate maximum about 12.00 M (Table 1). Values of k_1 for the perchloric acid-catalyzed hydrolysis of **4a–c** are shown in Figure 2.

The order of catalytic effectiveness of the acids observed for the hydrolysis of **4b** was $\text{HCl} \cong \text{H}_2\text{SO}_4 > \text{HClO}_4$ at low acidity. Bunton et al.^{12,13} suggested that such an order is characteristic of a bimolecular (A-2) mechanism, because the transition states of positive character are preferentially stabilized by anions of high charge density such as Cl^- , whereas the opposite is usually the case for an A-1 mechanism.

The kinetic data in Table 1 were also analyzed by the excess acidity treatment of Cox and Yates.¹⁴ A simplified version of their relationship for mainly unprotonated substrates

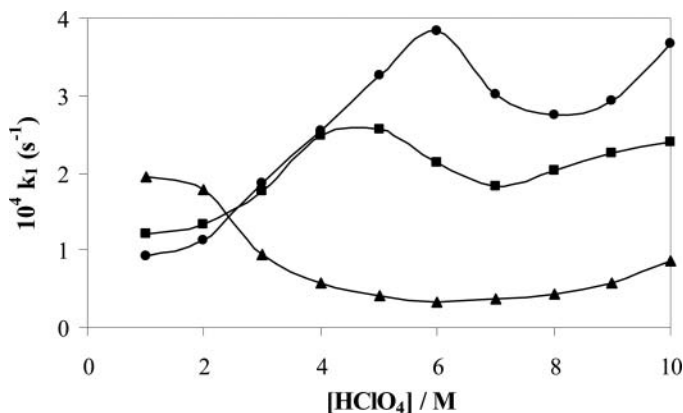


Figure 2 Plots of k_1 for the perchloric acid-catalyzed hydrolysis of **4a–c** at 40.0 °C; ●, p-CH₃; ■, p-H; ▲, p-Cl.

Table 1 Values of $10^4 k_1$ (s^{-1}) for the hydrolysis of N-(4-substituted arylthio) phthalimides at 40.0 ± 0.1 °C

Acid (M)	4a			4b			4c		
	H ₂ SO ₄	HClO ₄	HCl	H ₂ SO ₄	HClO ₄	HCl	H ₂ SO ₄	HClO ₄	HCl
0.50	—	—	1.03	—	—	2.25	—	1.04	1.08
1.00	1.66	1.22	1.40	4.26	0.93	2.36	2.34	1.41	1.47
2.00	1.57	1.43	1.49	4.00	1.13	2.63	1.89	1.48	1.75
2.50	—	—	—	—	—	2.86	—	—	—
3.00	1.46	1.86	1.25	3.66	1.86	3.30	1.22	0.94	1.64
3.50	—	—	—	—	—	3.88	—	—	—
4.00	1.29	2.49	1.11	3.16	2.55	4.02	0.81	0.58	1.27
4.50	—	—	—	—	—	3.52	—	—	—
5.00	1.05	2.56	1.08	2.49	3.27	3.06	0.47	0.41	1.03
5.50	—	—	—	—	3.72	—	—	—	—
6.00	0.72	2.14	1.17	1.38	3.84	2.51	0.23	0.33	1.11
6.50	—	—	—	—	3.57	—	—	—	—
7.00	0.54	1.82	1.32	0.61	3.01	2.37	0.28	0.37	1.27
8.00	0.49	2.03	1.63	0.39	2.75	2.54	0.37	0.44	1.52
9.00	0.68	2.23	2.15	0.56	2.94	2.77	0.53	0.6	1.94
10.00	1.35	2.41	2.98	0.99	3.68	3.12	0.71	0.87	2.55
11.00	1.87	—	3.82	1.72	—	3.93	0.81	—	3.62
11.50	—	—	—	2.64	—	—	—	—	—
12.00	2.03	—	—	3.04	—	—	0.94	—	—
12.50	1.80	—	—	2.86	—	—	—	—	—
13.00	0.95	—	—	2.23	—	—	0.84	—	—
14.00	0.35	—	—	1.56	—	—	0.29	—	—
15.00	0.28	—	—	1.37	—	—	0.13	—	—
15.50	—	—	—	1.98	—	—	—	—	—
16.00	1.13	—	—	3.23	—	—	0.24	—	—
17.00	2.85	—	—	4.69	—	—	0.44	—	—

(Eq. (3)) was used.

$$\log k_1 - \log C_{H^+} - \log Cs/(Cs + C_{SH^+}) = m^* m^\neq X + r \log a_{Nu} + \log (k_o/K_{SH^+}) \quad (3)$$

In Eq. (3), k_1 is the pseudo first-order rate constant, C_{H^+} is the concentration of aqueous acid, X is the excess acidity constant, and $m^* m^\neq$ is the combined slope parameter, where m^\neq is characteristic of the type of reaction (for A-1 processes $m^\neq > 1^{14}$; for A-2 processes $m^\neq \cong 1^{14}$), and m^* is obtained from protonation studies; $r \log a_{Nu}$ for the A-2 reaction represents the nucleophilic activity, where r is the number of water molecules involved in forming the transition state.

Due to the extremely low basicity of the N-(4-substituted arylthio)phthalimides studied, the protonation correction term [$\log Cs/(Cs + C_{SH^+})$] can be neglected. Values of X for the aqueous solution of the acid were used.^{15,16}

A plot of $\log k_1 - \log C_{H^+}$ versus X is shown in Figure 3 for the hydrolysis of **4b** in sulfuric acid solution. Similar graphs were observed for **4a** and **4c** in sulfuric acid solution. Initially, all such graphs for the arylthiophthalimides in the low-acidity region exhibit downward curvature, typical of A-2 reactions involving water.¹⁷

As can be seen in Figure 3, in the 1.00–8.00 M region with increasing acid concentration, the plot shows that the rate of reaction was reduced and there was no catalytic effect of the acids. With increasing acid concentration, the activation of water decreased

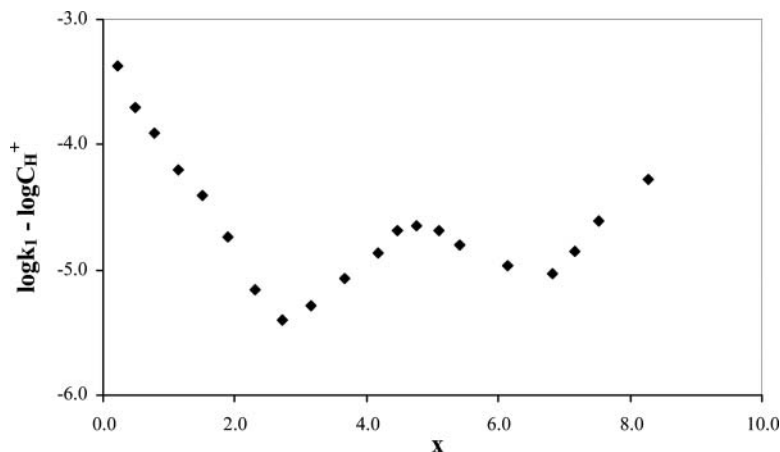


Figure 3 Plots of $\log k_1 - \log C_{H^+}$ versus excess acidity (X) for the sulfuric acid-catalyzed hydrolysis of **4b** at 40.0 °C.

and so the rate of reaction was reduced. After 8.00 M with increasing acid concentration, the rate of hydrolysis increased and there was a catalytic effect of the acids. A curve is observed in the 9.00–14.00 M region. A straight line was obtained in the acidity region (9.00–14.00 M); values of $3 \log a_{H_2O}$ can be subtracted from the left-hand side of Eq. (3) and the result plotted against X . A good straight line correlation (Figure 4 and Table 1 where $r = 3$) is obtained for arylthiophthalimides. These changes with increasing acidity to an upward linear region are characteristic of an A-1 process. This suggests that a gradual changeover in mechanism from A-2 to A-1 occurs with increasing concentration of acid.

Similar behavior has been observed for the hydrolysis of azophenyl ether,¹⁶ N-methyl-N-nitrobenzamides,¹⁸ benzohydroxamic acids,¹¹ and p-substituted phenyl N-(4-substituted arylamino)sulfonate esters.¹⁹

The temperature dependence of the rate constants of the hydrolysis reaction was analyzed by a least-squares procedure using a spreadsheet program (Eyring equation).

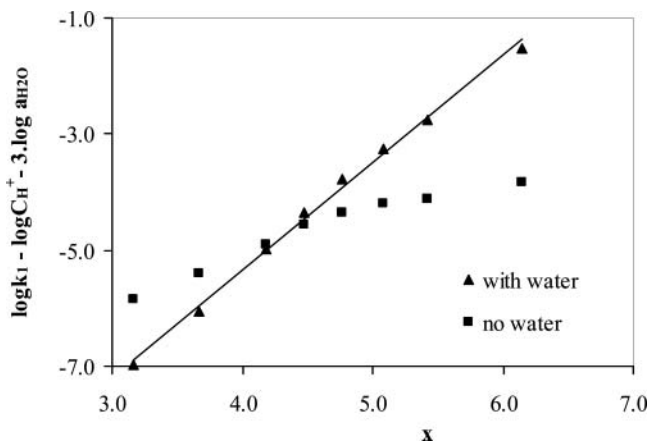


Figure 4 Plots of $\log k_1 - \log C_{H^+} - 3 \log a_{H_2O}$ versus excess acidity for the 9.00–14.00 M sulfuric acid-catalyzed hydrolysis of **4b** at 40.0 °C.

Table 2 Values of $10^4 k_1$ (s^{-1}) for the hydrolysis of **4a–c** at different temperatures

Acid (M)	Compounds	30.0 °C	35.0 °C	40.0 °C	45.0 °C	50.0 °C
1.00 M HClO ₄	4a	0.63	0.88	1.22	1.70	2.23
5.00 M HClO ₄		1.38	1.82	2.56	3.53	4.82
10.00 M HClO ₄		1.06	1.48	2.41	3.40	4.54
1.00 M HCl		0.69	0.94	1.40	1.92	2.41
5.00 M HCl		0.48	0.69	1.08	1.53	2.12
10.00 M HCl		1.17	2.05	2.98	4.90	7.38
1.00 M H ₂ SO ₄	4a	0.79	1.15	1.66	2.37	3.13
	4b	1.85	2.73	4.26	6.04	7.67
	4c	1.18	1.69	2.34	3.15	4.11
5.00 M H ₂ SO ₄	4a	0.52	0.77	1.05	1.75	2.38
	4b	0.99	1.55	2.49	3.45	5.00
	4c	0.21	0.33	0.47	0.65	0.91
10.00 M H ₂ SO ₄	4a	0.57	0.90	1.35	2.01	2.99
	4b	0.42	0.64	0.99	1.48	2.40
	4c	0.33	0.46	0.71	0.98	1.41
15.00 M H ₂ SO ₄	4b	0.53	0.88	1.37	2.19	3.49
	4c	0.056	0.083	0.130	0.194	0.296
16.00 M H ₂ SO ₄	4a	0.31	0.57	1.13	1.97	4.67
	4b	0.76	1.71	3.23	5.65	9.38
	4c	0.06	0.12	0.24	0.56	0.97

The values at different temperatures and Arrhenius parameters are shown in Tables 2 and 3, respectively. Acid-catalyzed hydrolyses of esters and amides²⁰ proceeding by an A-1 mechanism have ΔS^\ddagger of about 0 to $-41.8 \text{ JK}^{-1}\text{mol}^{-1}$, whereas those proceeding by an A-2 mechanism have ΔS^\ddagger of -62.8 to $-125.5 \text{ JK}^{-1}\text{mol}^{-1}$. Over the range 1.00–16.00 M

Table 3 Arrhenius parameters for the hydrolysis of N-(4-substituted arylthio) phthalimides

Compounds	Acid	[H ⁺]/M	ΔH^\ddagger (kJ/mol)	ΔS^\ddagger (J/molK)	Temperature Range (°C) ^a	R ²
4a	HClO ₄	1.00	51.99	−154.50	30–50	0.999
		5.00	51.51	−149.80	30–50	0.998
		10.00	60.98	−120.60	30–50	0.994
	HCl	1.00	52.43	−152.30	30–50	0.995
		5.00	61.39	−125.80	30–50	0.998
		10.00	74.23	−75.79	30–50	0.998
	H ₂ SO ₄	1.00	56.00	−137.10	30–50	0.999
		5.00	63.78	−120.30	30–50	0.994
		10.00	67.84	−104.20	30–50	0.999
	H ₂ SO ₄	16.00	108.45	+25.31	30–50	0.993
		1.00	59.36	−121.10	30–50	0.992
		5.00	65.86	−104.80	30–50	0.997
4b	H ₂ SO ₄	10.00	70.40	−97.35	30–50	0.998
		15.00	76.25	−75.92	30–50	0.999
		16.00	101.50	+11.08	30–50	0.994
4c	H ₂ SO ₄	1.00	50.84	−152.90	30–50	0.999
		5.00	58.74	−141.10	30–50	0.997
		10.00	59.63	−134.80	30–50	0.998
		15.00	68.06	−121.80	30–50	0.999
		16.00	115.77	+36.09	30–50	0.997

^aMeasurements were made at 5 °C intervals.

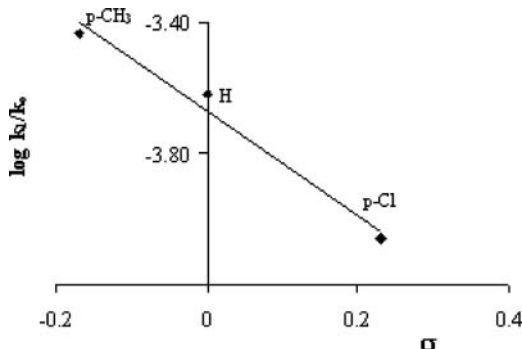


Figure 5 The plot of $\log k_1$ versus Hammett σ values for acid-catalyzed hydrolyses (10.00 M HClO_4) of N-(4-substituted arylthio)phthalimides at 40.0 °C.

sulfuric acid the values of ΔS^\ddagger changed from -137.10 to $+25.31$, -121.10 to $+11.08$, and -152.90 to $+36.09$ $\text{JK}^{-1}\text{mol}^{-1}$ for the hydrolysis of **4a**, **4b**, and **4c**, respectively. Similar behavior has been observed for the acid-catalyzed hydrolysis of (4-methoxybenzoyl)-4-tolueniminosulfonate.²¹ Values of ΔS^\ddagger for the hydrolysis of **4a–c** become increasingly less negative or positive with increased acidity, suggesting a mechanistic changeover from A-2 to A-1. This suggests an A-2 mechanism at low acid concentrations and an A-1 mechanism at high acid concentrations.

In the acidity range studied, electron-donating substituents cause the highest rate of hydrolysis (**4b** > **4c**), and the substituent effects are well correlated by a satisfactory Hammett $\rho\sigma$ plot (at 10.00 M HClO_4 , $\rho = -1.586$ [corr. 0.978]) as shown in Figure 5. At these acidities electron-donating substituents both facilitate protonation of the substrate and stabilize the sulfur cation in the case of an A-1 mechanism. At lower acidities, however (e.g., 1.00 M HClO_4 , $\rho = 0.803$ [corr. 0.996]) as shown in Figure 6, **4c** hydrolyzes more rapidly than **4b**, consistent with a predominantly A-2 mechanism, in which substituent effects on the protonation and slow step operate in opposite directions. There is no direct evidence concerning the site of protonation of N-(4-substituted arylthio)phthalimides;

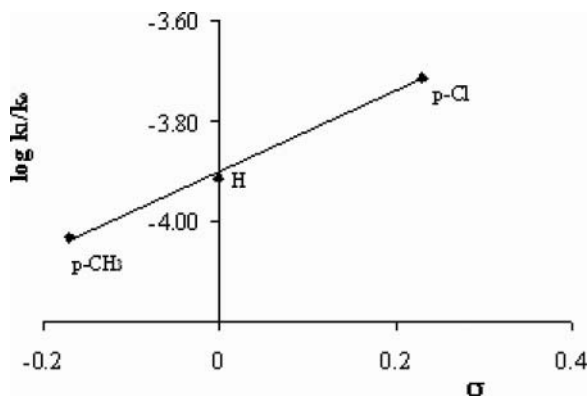
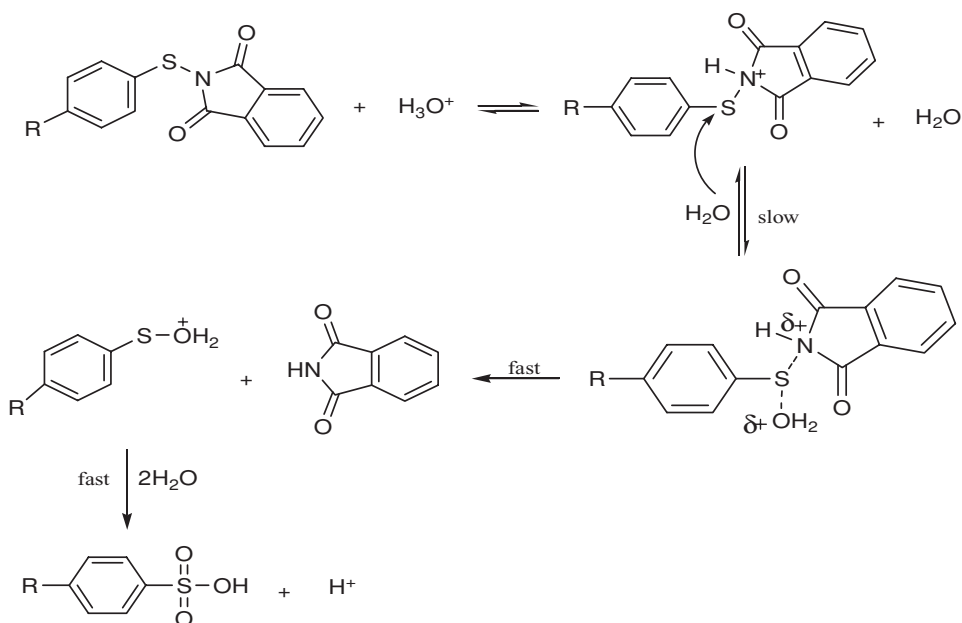


Figure 6 The plot of $\log k_1$ versus Hammett σ values for acid-catalyzed hydrolyses (1.00 M HClO_4) of N-(4-substituted arylthio)phthalimides at 40.0 °C.



Scheme 2

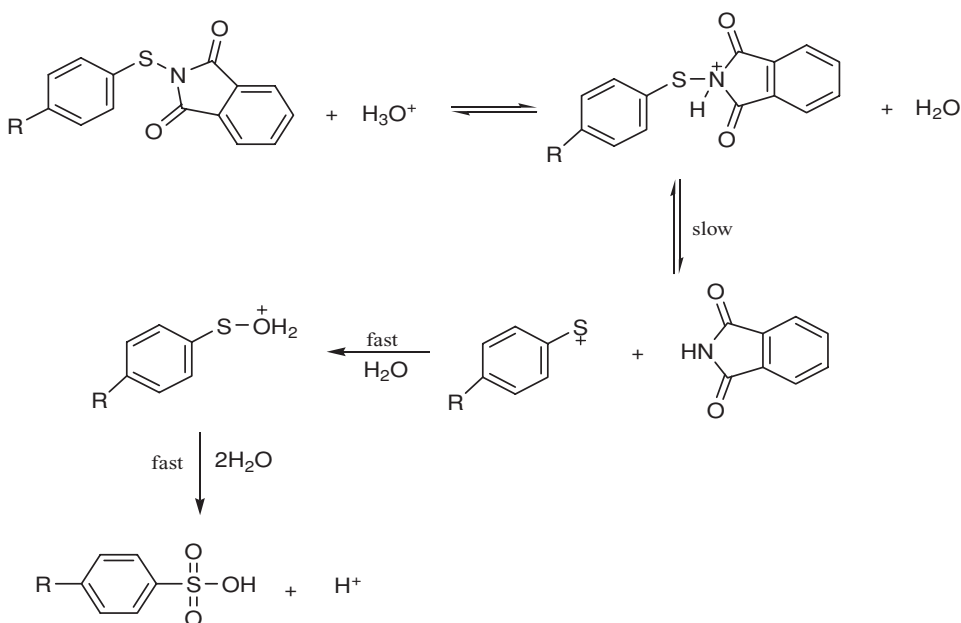
however, protonation of sulfinamides,^{10,22} sultams,²³ and cyclic sulfamate esters²⁴ occurs preferentially at the nitrogen atom.

In light of the overall evidence, we propose that the acid-catalyzed hydrolysis of N-(4-substituted arylthio)phthalimides occurs according to an A-2 mechanism at lower acidities and an A-1 mechanism at higher acidities, as shown in Schemes 2 and 3, respectively. Sulfinic acids are known to be readily oxidized; for instance, p-toluenesulfinic acid undergoes facile autoxidation in acetonitrile at 40.0 °C to give p-toluenesulfonic acid and the autoxidation is accelerated by the presence of a Cl^- or Cu(II) ion.²⁵

EXPERIMENTAL

Materials

N-(4-Substituted arylthio)phthalimides **4a–c** were prepared from the corresponding 4-substituted thiophenol by reaction with phthalimide in hot acetonitrile and pyridine and then a solution of bromine in acetonitrile, as described by Klose et al.²⁶ $^1\text{H-NMR}$ spectra were recorded on a Bruker AC 200 MHz spectrometer using tetramethylsilane (TMS) as an internal standard. Infrared spectra were recorded on a Vertex 80v Fourier transform infrared (FTIR) spectrometer. All melting points were obtained using an electrothermal digital melting point apparatus. **4a**: m.p. 161–163 °C (lit.²⁶ 160–161 °C); **4b**: m.p. 204–205 °C. Found C, 67.60; H, 4.28; N, 5.28; S, 11.88. Calcd. for $\text{C}_{15}\text{H}_{11}\text{NO}_2\text{S}$: C, 66.89; H, 4.12; N, 5.20; S, 11.91%; **4c**: m.p. 179–181 °C (lit.²⁶ 179–181 °C).



Scheme 3

Kinetic Studies

The rates of hydrolysis of N-(4-substituted arylthio)phthalimides were followed spectrophotometrically at 225–240 nm using a GBC Cintra 20 model ultraviolet-visible (UV-Vis) spectrophotometer (Australia) with a thermostated cell compartment ($\pm 0.05^\circ\text{C}$). Good first-order behavior was observed with clean isosbestic points. Values of k_1 were calculated from the standard equation using a least-squares procedure. All kinetic measurements were duplicated and the average deviation from the mean was $<3\%$.

Product Analysis

The products of hydrolysis were determined by comparing the UV spectrum obtained after completion of the kinetic experiment with the spectrum of the expected products at the same concentration and under the same conditions. Thus, for the hydrolysis of N-(4-methylphenylthio)phthalimide, the UV spectrum recorded at the end of the reaction was identical with that of a 1:1 mixture of phthalimide and *p*-toluenesulfonic acid. Phthalimide was obtained from Fluka (Buchs SG, Switzerland) and *p*-toluenesulfonic acid was prepared as described in the literature.²⁷

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