≡ S_{Cl_2}), then again with N_2 as the dominant quencher. Signals were normalized for $[O_3]$ and laser power. In addition, it was necessary to correct S_{Cl_2} for absorption of resonance lamp radiation by Cl_2. The procedure for making this correction is described elsewhere.[10] At the highest Cl_2 concentration employed, absorption of resonance lamp radiation resulted in a 25% drop in signal count rate. Since it is known that $O(^1D)$ deactivation by N_2 produces $O(^3P)$ with unit yield, k_{2c}/k_2 could be determined from the relationship

$$k_{2c}/k_2 = \{(S_{Cl_2}/S_{N_2}) - \Phi\}(1 - \Phi)^{-1} \qquad (X)$$

where Φ is the quantum yield for producing $O(^3P)$ from O_3 photolysis at 248 nm, a quantity which we have previously measured to be $0.093 \pm 0.028[\beta^*/(1 + \beta^*) = \Phi]$.[10] Fourteen determinations of S_{Cl_2}/S_{N_2} gave an average value of 0.323 with $2\sigma = 0.060$. According to eq X, we obtain for k_{2c}/k_2 the value 0.254 ± 0.097 where the quoted error is 2σ and includes uncertainties in both S_{Cl_2}/S_{N_2} and Φ. Two points are worth noting regarding this result. First, it is unlikely that secondary chemistry involving Cl, ClO, or photolytically produced $O_2(^1\Delta)$ could have influenced the measurement of k_{2c}/k_2; in order to be important, secondary chemistry would have had to occur on a time scale of a few hundred microseconds. Also, in addition to our measurement of Φ, Greenblatt and Wiesenfeld[15] have determined Φ to be 0.06 ± 0.01. Using their value in eq X would have only a minor effect on the result, changing k_{2c}/k_2 from 0.254 to 0.280.

Weighting the more precise of the two independent determinations more strongly, we report $k_{2c}/k_2 = 0.25 \pm 0.10$. Reaction 2 has generally been assumed to proceed exclusively via the reactive channel 2a.[7,16] Our results, however, indicate that the physical quenching channel is significant.

There is one absolute determination of k_2 reported in the literature. Fletcher and Husain,[7] employing flash photolysis in conjunction with time-resolved resonance absorption detection of $O(^1D)$, obtained the value $k_2 = (2.2 \pm 0.2) \times 10^{-10}$ cm^3 molecule^{-1} s^{-1}. Husain and co-workers have studied a large number of $O(^1D)$ reactions and, in nearly all cases, report rate coefficients which are about a factor of 2.2 larger than those measured by other experimental techniques; this systematic difference is presumably due to incorrect determination of γ, a parameter which relates the absorbance to the $O(^1D)$ concentration. Interestingly, our value for k_2 is about 25% faster than the value reported by Fletcher and Husain. The reason for this deviation from the trend observed for other reactions is unclear at this time. In addition to Fletcher and Husain's measurement, Freudenstein and Biedenkapp[8] have employed flash photolysis in conjunction with kinetic absorption spectroscopic detection of ClO to obtain a measurement of k_{2a}, the rate coefficient for the reactive channel which produces ClO, relative to the rate coefficient for the process

$$O(^1D) + O_3 \rightarrow \text{products} \qquad (7)$$

Their result was $k_{2a}/k_7 = 0.75 \pm 0.25$. Using our value for $k_{2a} + k_{2b}$, $(2.11 \pm 0.34) \times 10^{-10}$ cm^3 molecule^{-1} s^{-1}, in conjunction with the recommended value[12] for k_7, gives $(k_{2a} + k_{2b})/k_7 = 0.88$. Our results are consistent with those of Freudenstein and Biedenkapp. Although it appears that $k_{2a} \gg k_{2b}$, the combined uncertainties of the two studies are large enough that a significant rate for a channel 2b cannot be completely ruled out.

Acknowledgment. This work was supported by the Fluorocarbon Program Panel of the Chemical Manufacturers Association.

Registry No. O_2, 7782-44-7; Cl_2, 7782-50-5.

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One-Electron Redox Reactions in Aqueous Solutions of Sulfite with Hydroquinone and Other Hydroxyphenols

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The one-electron oxidation of hydroquinone and other hydroxyphenols by the sulfite radical, $\dot{S}O_3^-$, and the peroxysulfate radical, $\dot{S}O_5^-$, has been investigated along with the oxidation of sulfite by several phenoxyl radicals. Rate constants for the oxidation of hydroxyphenols by $\dot{S}O_3^-$ are small in neutral solution but increase dramatically in basic solution, in correspondence to the deprotonation of the hydroxyphenol. $\dot{S}O_5^-$ reacts with most hydroxyphenols at moderate rates ($k \sim 1$-5×10^6 M^{-1} s^{-1}) in neutral solution. The phenoxyl radicals with higher redox potentials, from phenol, resorcinol, and phloroglucinol, were found to oxidize sulfite in neutral solutions.

In 1882, Berkely discussed the use of sulfite as a preservative in hydroquinone-based developer solutions,[1] where it inhibits hydroquinone autoxidation.[2] The use of hydroquinone and other phenolic compounds to inhibit sulfite autoxidation was demonstrated more recently.[3-6] It was shown that in the hydroquinone–sulfite system, mono- and disulfonates were formed in the early stages of the reaction.[7] Since phenolic substances and many other organic compounds also inhibit the photoinduced oxidation of sulfite,[8-10] it was suggested that the inhibition was due to their scavenging free radicals and therefore breaking the chain reaction.[10] Studies on the comparative effect of phenolic inhibitors show a rough correlation between their effectiveness and their two-electron redox potentials.[11,12]

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In order to understand better the mechanism of the autoxidation of sulfite solutions and the chemical effects associated with this reaction, we have undertaken a kinetic study of radical reactions involving sulfite and several phenolic compounds. Using a pulse radiolysis technique, we have measured rate constants for $\dot{S}O_3^-$ and $\dot{S}O_5^-$ radical reactions with phenols at several pH values and also the rates of reaction of sulfite with some phenoxyl radicals.

Experimental Section[13]

Sodium sulfite and bisulfite were analytical grade reagents from Fisher and Mallinckrodt, respectively. The amount of S(IV) in the sodium bisulfite was determined by dissolving a sample in 6 M $HClO_4$ and measuring the absorbance at 280 nm. An SO_2 absorptivity $\epsilon_{280} = 367$ M^{-1} cm^{-1} was used.[14] The phenolic compounds were from Aldrich and were used as received, except for hydroquinonemonosulfonate which was recrystallized from water. Water was purified by a Millipore Milli-Q system and solutions were freshly prepared each day. The pH was adjusted where necessary by using potassium or sodium hydroxide or perchloric acid, or was maintained by using phosphate or borate buffers. Solutions were bubbled to saturation with N_2O and flowed through an irradiation cell with a 2-cm optical path perpendicular to the electron beam. The flow rate was sufficient to replenish the solution in the cell between pulses.

The pulse radiolysis apparatus[15] consists of a Febetron 705 accelerator supplying 50-ns pulses of 2-MeV electrons. The dose per pulse was usually 500 rd, determined by KSCN dosimetry. The optical detection system used a Varian 300-W Xenon lamp, separated from the cell by a shutter, and a monochromator and photomultiplier located in a separate room from the irradiation region. The kinetic traces were digitized by a Tektronix 7612 transient recorder and process by a PDP 11/34 minicomputer. First-order rate constants were derived using a linear least-squares routine. Single-shot transient spectra were obtained using a EG&G PAR optical multichannel analyzer with a silicon photodiode array coupled to a Jarrell-Ash Mark X spectrograph.

Radiolysis of N_2O saturated aqueous solutions at pH 3–13 produces the hydroxyl radical predominantly. This radical reacts with sulfite or bisulfite ions to form the sulfite radical very rapidly

$$OH + SO_3^{2-} \rightarrow OH^- + \dot{S}O_3^- \quad (1)$$

$$OH + HSO_3^- \rightarrow H_2O + \dot{S}O_3^- \quad (2)$$

$k_1 = 5.5 \times 10^9$ and $k_2 = 9.5 \times 10^9$ M^{-1} s^{-1}.[16] The hydroxyl radical reacts also with the phenolic compounds with diffusion-controlled rate constants ($k \sim 10^{10}$ M^{-1} s^{-1})[17] to form initially OH adducts which then lose water or OH^- to yield the phenoxyl radical, e.g.,

$$\dot{O}H + C_6H_5OH \rightarrow HO\dot{C}_6H_5OH \rightarrow C_6H_5\dot{O} + H_2O \quad (3)$$

This reaction competes effectively with reactions 1 and 2 and, therefore, the concentrations of sulfite and of the aromatic compound have to be adjusted in order to favor the desired reaction. The hydrogen atoms, produced in small yield (~10% of OH yield) during the radiolysis, do not interfere in these systems since they do not react with sulfite and their reaction with the phenols yields H adducts that absorb at different wavelengths from the phenoxyl radicals.

In certain experiments, OH radicals were allowed to react with excess Br^- to form $Br_2^-\cdot$ radicals, which subsequently oxidized either sulfite or the phenol, depending on their relative concentrations. (In the course of this work we measured $k = 7 \times 10^7$ M^{-1} s^{-1} for $Br_2^-\cdot$ + hydroquinone at pH 6.7.)

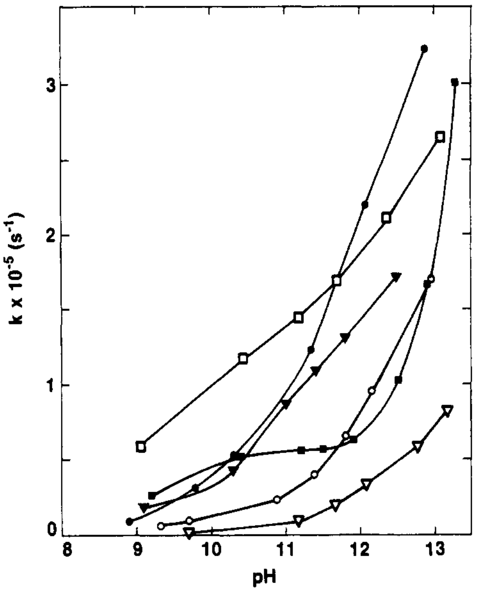

Figure 1. First-order rate constant for the reaction of $\dot{S}O_3^-$ with hydroxyphenols as a function of pH, measured at a hydroxyphenol concentration of 1–3 mM and normalized to 1 mM: ●, hydroquinone; ■, catechol; ▼, resorcinol; □, pyrogallol; ○, hydroquinone-2-sulfonate; ▽, hydroquinone-2,5-disulfonate.

TABLE I: pK_a Values and Redox Potentials for Phenolic Compounds

compd	pK_1[a]	pK_2[a]	$E^1_{13.5}$[b]	E^1_7[b]	radical λ_{max}
phenol	10.0		0.7	0.9	402[c]
resorcinol	9.3	11.2	0.39	0.81	450[d]
catechol	9.4	13.0	0.04	0.53	300[e]
hydroquinone	9.9	11.5	0.02	0.46	430[f]
pyrogallol	9.1	11.3	−0.01		~320[g]
phloroglucinol	8.0	9.2			
hydroquinone-2-sulfonate					430
hydroquinone-2,5-disulfonate			0.12[h]		450

[a] From: Serjeant, E. P.; Dempsey, B. "Ionization Constants of Organic Acids in Aqueous Solution"; Pregamon Press: Oxford, 1979. [b] One-electron redox potentials (V vs. NHE) for the couples $Ph\dot{O}/PhO^-$ or the corresponding analogues, depending on compound and pH, taken from: Steenken, S.; Neta, P. *J. Phys. Chem.* **1982**, *86*, 3661, except for the value for phenol which is from ref 18. [c] From: Schuler, R. H.; Buzzard, G. K. *Int. J. Radiat. Phys. Chem.* **1976**, *8*, 563. [d] From Steenken and Neta, above. [e] From: Adams, G. E.; Michael, B. D. *Trans. Faraday Soc.* **1967**, *63*, 1171. [f] From: Patel, K. B.; Willson, R. L. *J. Chem. Soc., Faraday Trans. 1* **1973**, *69*, 814, and others. [g] By comparison with substituted derivatives (Steenken and Neta, above). [h] pH 12.9.

The peroxysulfate radical, $\dot{S}O_5^-$, is produced by the reaction of $\dot{S}O_3^-$ with oxygen ($k \sim 1.5 \times 10^9$).[18] For the study of $\dot{S}O_5^-$ reactions with phenols we have saturated the aqueous sulfite–phenols solutions with a mixture of N_2O:O_2 = 1:1.

Results

Reactions of $\dot{S}O_3^-$. Inhibition of sulfite autoxidation by phenolic compounds is believed to be due to the oxidation of these compounds by radical intermediates. The initial radical produced upon one-electron oxidation of sulfite is $\dot{S}O_3^-$. Recently,[18-20] we have reported rate constants for the oxidation of several organic compounds by $\dot{S}O_3^-$ and have found this radical to be a mild oxidant. In the present study we have examined the rates of oxidation of the various phenolic compounds by $\dot{S}O_3^-$. We find that in neutral

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[13] Certain commercial equipment, instruments, or materials are identified in this paper in order to specify adequately the experimental procedure. Such identification does not imply recognition or endorsement by the National Bureau of Standards, nor does it imply that the material or equipment identified are necessarily the best available for the purpose.

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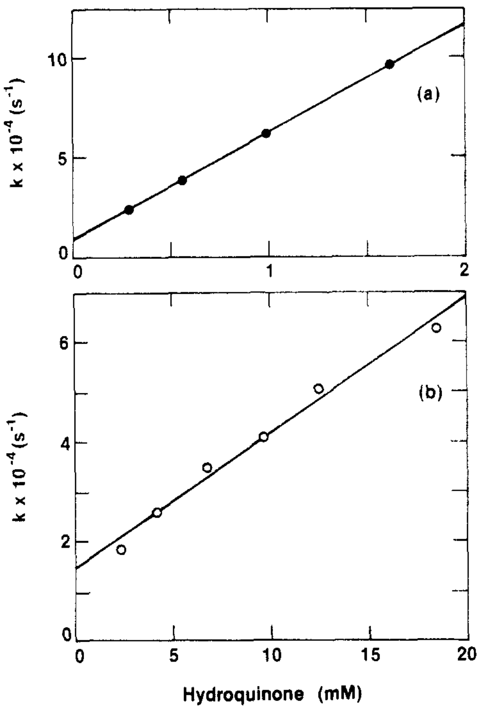

Figure 2. Concentration dependence of the first-order rate constant for the reactions of hydroquinone with $\dot{S}O_3^-$ at pH 10.5 (a) and with $\dot{S}O_5^-$ at pH 6.6 (b).

solutions the oxidation of these compounds by $\dot{S}O_3^-$ is extremely slow and could not be observed under the pulse radiolysis conditions. In alkaline solutions the phenolic compounds deprotonate

$$HO{-}C_6H_4{-}OH \xrightleftharpoons{K_1} HO{-}C_6H_4{-}O^- + H^+ \quad (4)$$

$$HO{-}C_6H_4{-}O^- \xrightleftharpoons{K_2} {}^-O{-}C_6H_4{-}O^- + H^+ \quad (5)$$

and the anions are known to be far more reactive toward oxidation than the neutral species. Indeed, we find the rate of oxidation by $\dot{S}O_3^-$ to increase dramatically with pH in a manner consistent with the pK_a's of the compounds. In Figure 1 we present these rate constants normalized to 1 mM phenolic compound for comparison. It is difficult to compare the rate constants for the various phenols quantitatively at any particular pH due to the effect of the equilibria 4 and 5. In general, the pK_a's of the compounds are too close (Table I) to allow experimental separation of k_6

$$\dot{S}O_3^- + HOC_6H_4O^- \rightarrow SO_3^{2-} + {}^-OC_6H_4\dot{O} + H^+ \quad (6)$$

$$\dot{S}O_3^- + {}^-OC_6H_4O^- \rightarrow SO_3^{2-} + {}^-OC_6H_4\dot{O} \quad (7)$$

and k_7, with the notable exception of catechol. Even in the latter case it was difficult to obtain a good fit with a calculated pK curve due to the effect of ionic strength. Nevertheless, the correlation between the rate constants and the pK_a values is obvious. At the high pH limit, hydroquinone, catechol, and pyrogallol have somewhat similar redox potentials (Table I) and all appear to react with $\dot{S}O_3^-$ very rapidly. Resorcinol reacts slightly more slowly due to its higher potential. The hydroquinonesulfonates, on the other hand, appear to react more slowly partly because of the electrostatic effects of the additional negative charges and partly because of higher potential (see below).

For hydroquinone and resorcinol we carried out a more detailed measurement of the rate constant at a particular pH. For hydroquinone at pH 10.5 (Figure 2a) we find $k = 5.4 \times 10^7$ M^{-1} s^{-1} and for resorcinol at pH 12.5 $k = 1.5 \times 10^8$ M^{-1} s^{-1}.

At pH >7, the redox potential for the $\dot{S}O_3^-/SO_3^{2-}$ couple is 0.63 V vs. NHE.[18] Therefore, even at pH 7 $\dot{S}O_3^-$ is thermodynamically able to oxidize catechol and hydroquinone (E given in Table I). It has been observed, however, that for even stronger oxidants, the rate constants for the oxidation of the phenols are very slow at pH 7,[21,22] so the lack of reactivity of $\dot{S}O_3^-$ is not surprising.

TABLE II: Rate Constants for Oxidation of Sulfite by Phenoxyl Radicals

phenol	pH	k, M^{-1} s^{-1}
phenol	11	1×10^7
resorcinol	7	2.3×10^6
phloroglucinol	7	3.2×10^6
hydroquinone-2,5-disulfonate	7	$<10^4$

TABLE III: Rate Constants for Oxidation of Phenols by $\dot{S}O_5^-$ Radicals

compd	pH	k, M^{-1} s^{-1}
pyrogallol	6.7	4.1×10^6
catechol	6.7	2.7×10^6
hydroquinone	6.6	2.7×10^6
	9.5	2.0×10^7
hydroquinone-2-sulfonate	6.8	1.5×10^6
hydroquinone-2,5-disulfonate	6.6	8.2×10^5
	9.1	4.7×10^6
resorcinol	6.7	$<1 \times 10^6$

For resorcinol and phenol at pH 7, the reaction is expected to be reversed, based on their redox potentials.

$${}^-O{-}C_6H_4\dot{O} + SO_3^{2-} + 2H^+ \rightarrow HOC_6H_4OH + \dot{S}O_3^- \quad (8)$$

Table II gives values of k_8 for phenol, resorcinol, and phloroglucinol. The latter two would be expected to behave similarly since both of their radicals are of a *m*-semiquinone type which lack resonance stabilization. Reaction 8 was found to be too slow to measure for hydroquinone-2,5-disulfonate and is expected to be even slower for the monosulfonate.

For hydroquinone at pH 7 reaction 8 was found to be too slow to measure in either direction. However, when the semiquinone and the $\dot{S}O_3^-$ radicals were both produced concurrently, their mutual reaction was apparent as an increase in the decay rate of the semiquinone radical. Reaction 9 may proceed by addition

$${}^-OC_6H_4\dot{O} + \dot{S}O_3^- \rightarrow \text{products} \quad (9)$$

of $\dot{S}O_3^-$ to a ring carbon to form hydroquinonesulfonate. This process may be important in the hydroquinone–sulfite system, where inhibition of autoxidation was found to be accompanied by sulfonation of the hydroquinone.

Reactions of $\dot{S}O_5^-$. The other major fate of $\dot{S}O_3^-$ during autoxidation is to react with O_2.

$$\dot{S}O_3^- + O_2 \rightarrow \dot{S}O_5^- \quad (10)$$

In previous work we found $\dot{S}O_5^-$ to be a stronger oxidant than $\dot{S}O_3^-$.[18-20] The potential of $\dot{S}O_5^-$ was estimated to be ~1.1 V at pH 7, which is higher than those of the phenolic compounds (Table I). This suggests that the electron-transfer reaction 11 is favorable.

$$\dot{S}O_5^- + HOC_6H_4OH \rightarrow HSO_5^- + {}^-OC_6H_4\dot{O} + H^+ \quad (11)$$

Therefore, we have examined the reactivity of $\dot{S}O_5^-$ with the phenolic compounds at pH 7. Table III shows that these rate constants are moderate at pH 7 and increase at higher pH. In general, the phenolic compounds are unstable at high pH in the presence of oxygen (even in the presence of sulfite) and, therefore, detailed study of $\dot{S}O_5^-$ reactions as a function of pH was not possible. To ensure the reliability of the rate constants in Table III they were measured at several concentrations as exemplified in Figure 2b. The order of reactivities in Table III is as expected from the redox potentials, although for the sulfonated compounds

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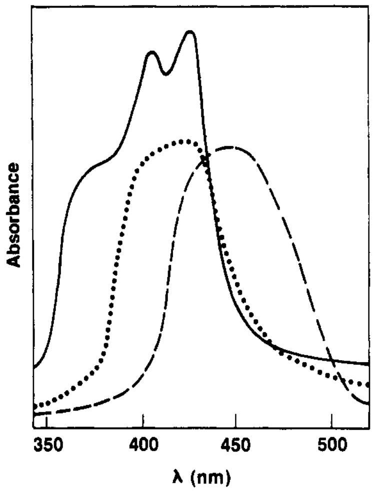
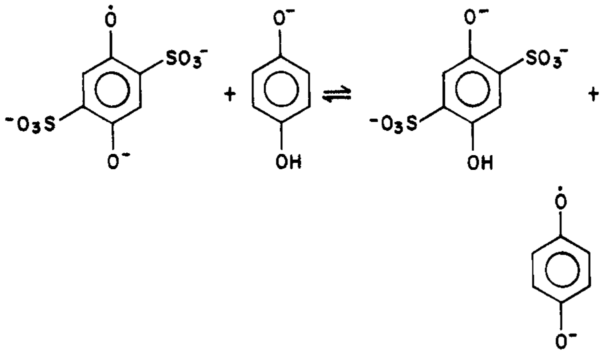


Figure 3. Absorption spectra of the semiquinone radicals from hydroquinone (solid line), hydroquinone-2-sulfonate (dotted line), and hydroquinone 2,5-disulfonate (dashed line).

the additional negative charge may decrease the reactivity due to electrostatic repulsion.

Properties of Sulfonated Semiquinones. Although the spectra of the other semiquinone radicals have been measured previously (see references in Table I) there are no reported spectra on their sulfonated derivatives. Therefore, we have determined the spectra of the mono- and disulfonated *p*-semiquinone in comparison with the unsubstituted radical (Figure 3). Since the spectrum of the disulfonated derivative is red shifted from that of semiquinone, we used this difference to follow the electron transfer between these two compounds.



(12)

At pH 12.9 an equilibrium was established with $K_{12} \approx 37$, $k_{12} = 7.8 \times 10^5$ M^{-1} s^{-1}, and $k_{-12} \sim 2.1 \times 10^4$ M^{-1} s^{-1}. This gives $\Delta E \approx 93$ mV between hydroquinone and the disulfonate. By using $E^1 = 0.023$ V for hydroquinone at this pH,[22] we calculate $E^1 = 0.116$ V for the disulfonate.

Discussion

The order of inhibition for the Mn(II) catalyzed oxidation of a sodium sulfite solution (pH ~9) was reported to be pyrogallol > hydroquinone > catechol > phloroglucinol > resorcinol > phenol.[12] The order for a sodium bisulfite solution with no catalyst specified was reported to be hydroquinone > pyrogallol > catechol > resorcinol > phenol > phloroglucinol.[11] At pH 7.2, hydroquinone and its mono- and disulfonate derivatives were all reported to have about the same inhibiting effect,[2] although other authors suggest that the sulfonates are better inhibitors.[7]

These results are not inconsistent with our observations on the relative reactivity of these compounds toward $\dot{S}O_3^-$ and $S\dot{O}_5^-$, but it is unlikely that this alone is sufficient to explain the influence of phenolic compounds on sulfite autoxidation. One other possible effect, the formation of metal–inhibitor complexes with *o*-hydroxy derivatives has been noted previously.[12]

In addition to these phenolic species many other organic compounds are known to inhibit the autoxidation of sulfite, frequently with the concomitant chemical transformation of the inhibitor.[23] In general, it appears that the more powerful inhibitors, such as these phenolic species, are reactive toward $\dot{S}O_3^-$ or $S\dot{O}_5^-$, whereas the less powerful inhibitors, such as aliphatic alcohols, are unreactive. For these latter compounds, the inhibition mechanism must involve more reactive radicals, most likely $S\dot{O}_4^-$.[23] Reaction with this radical could also play a role in the inhibition of sulfite autoxidation by phenolic compounds. Since the reactivity of $S\dot{O}_4^-$ with all of the phenolic compounds is very high, this reaction would not lead to the observed variation in inhibition among these compounds. The variation, however, could arise from the differences in the rates of reaction of the phenoxyl radicals with sulfite. In other words, either an increase in k_6 or k_7 or a decrease in k_8 will increase the effectiveness of the inhibitor.

Acknowledgment. The research described herein was supported in part by the Office of Basic Energy Sciences of the Department of Energy.

Registry No. SO_3^{2-}, 14265-45-3; $\dot{S}O_3^-$, 12210-38-7; $S\dot{O}_5^-$, 12509-93-2; pyrogallol, 87-66-1; catechol, 120-80-9; hydroquinone, 123-31-9; hydroquinone-2-sulfonate, 68864-98-2; hydroquinone-2,5-disulfonate, 97190-49-3; resorcinol, 108-46-3; phenol, 108-95-2; phloroglucinol, 108-73-6; phenol (phenoxy radical), 2122-46-5; resorcinol (phenoxy radical), 322-29-4; phloroglucinol (phenoxy radical), 69751-57-1; hydroquinone-2,5-disulfonate (phenoxy radical), 56534-12-4.

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