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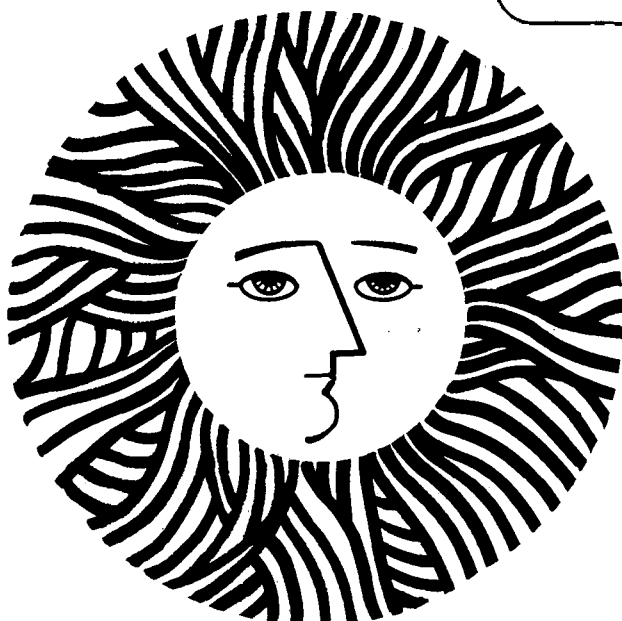
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August 1981

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SOOT-CATALYZED ATMOSPHERIC REACTIONS*

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Abstract

This paper reviews the work of the Atmospheric Aerosol Research group at Lawrence Berkeley Laboratory on soot-catalyzed atmospheric reactions. Data are presented on the soot concentrations in various geographic regions of the United States. The experimental results on SO₂ oxidation on both dry and wet soot particles are reviewed.

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Introduction

Atmospheric aerosol particles are classified as primary or secondary, depending on their origin. Primary particles are produced by sources such as combustion devices and are introduced into the atmosphere in particulate form. Secondary particles are formed in the atmosphere by chemical reactions among primary and secondary gaseous species, primary particles, and gaseous and liquid water.

Depending on the phases of the reactants, atmospheric reactions can be homogeneous or heterogeneous. Homogeneous reactions involve only gaseous species, while heterogeneous processes may involve gases and solid particles, gases and liquid droplets, or three-phase systems with gases, liquid droplets, and solid materials occluded in these droplets. These heterogeneous processes may be catalytic or stoichiometric; they may proceed in the bulk of a droplet, on the gas-solid interface, or on the solid-liquid interface.

In this paper we shall discuss the last two categories, specifically in conjunction with the role of combustion-generated soot particles in the oxidation of sulfur dioxide. Soot is synonymous with primary carbonaceous particulate material; it is a chemically complex material consisting of an organic component and a component variously referred to as elemental, graphitic, or black carbon; but for consistency we shall use the term black carbon here. Soot and smoke were the first air pollutants to be recognized and controlled. The word "smog" is a contraction of "smoke" and "fog." Such smog became known as London-type smog.

Soot in the atmosphere not only contributes to the total particulate concentration in ambient air but also may serve as an efficient catalyst for atmospheric reactions such as the oxidation of SO_2 to sulfate. Soot has properties similar to those of activated carbon, which is well known to be a

catalytically and surface chemically active material.

While at one time the presence of soot in the atmosphere of industrial cities was obvious, it became less obvious in more recent times. Improvements in combustion technology and the use of better-grade fuels have led to the virtual elimination of visible smoke emissions. The emphasis of air pollution control thus shifted away from primary particulate emissions toward controlling gaseous emissions, especially in view of the newer concept of Los Angeles-type photochemical smog, which was believed to contain neither smoke nor fog. According to such a view, the haze over the Los Angeles Air Basin on polluted days is due almost entirely to the photochemical conversion of certain invisible gases to light-scattering particles consisting of sulfates, nitrates, and secondary organics but almost no soot.

The assessment of the chemical role of soot in the atmosphere in general, and in photochemical environments such as Los Angeles in particular, had to start with an empirical assessment of the soot concentrations. The results of these studies, as shown below, have clearly demonstrated that soot is ubiquitous not only in urban atmospheres but also in remote regions such as the Arctic.¹ Therefore Los Angeles, with its abundant coastal fog, contains both components of London-type fog — smoke (or soot) and fog.

Soot in the Atmosphere

Our first indication of the presence of soot in the Los Angeles atmosphere came from photoelectron spectroscopy (ESCA) of ambient samples.² These spectra showed that most of the particulate carbon is in a neutral chemical state compatible with combustion-generated soot. According to the photochemical hypothesis, the secondary organics should be oxygenated, resulting in an easily detectable chemical shift of the ESCA carbon peak. In our spectra, only a very small fraction of the carbon peak of the ambient samples was found to be chemically

shifted. With ESCA we also found that the ambient carbonaceous material is relatively nonvolatile when the samples were heated to 350°C in vacuum. Such measurements, combined with visual observation of the filter samples (all of which appeared black or grey), led us to postulate that a substantial fraction of the total particulate carbon in the Los Angeles basin is in the form of soot. In recent years, we initiated a field measurement program to identify and quantify the amount of soot in many locations across the United States.

The principal approach used in our laboratory relies on the use of black carbon as a tracer for primary carbonaceous^{3,4} material because black carbon can be produced only in a combustion process and is therefore definitely primary. The methodology that we adopted involved systematic measurements of the ratio of black carbon to total carbon for a large number of samples collected directly from sources, source-dominated environments, and well-aged ambient air (24-hour samples).⁵ The ambient samples were collected in areas with widely different atmospheric chemical characteristics (e.g., degree of photochemical activity, source composition, geographic location). Measurements of this ratio from a number of source samples give insights into the relative black to total carbon ratio of primary emissions and the source variabilities. Secondary material will not contain the black component but will increase the total mass of carbon and therefore reduce the black to total carbon fraction. That is, under high photochemical conditions one would expect this ratio to be significantly smaller than under conditions obviously heavily influenced by sources.

Because of the large number of samples that had to be analyzed, a fast-throughput optical attenuation method³ was used for determining black carbon. The validity of the optical attenuation method was checked by performing Raman spectroscopic⁶ and opto-acoustic⁷ measurements on some of the ambient and

source samples. Total particulate carbon was determined by a combustion method.

The optical attenuation method compares the transmission of a 633-nm He-Ne laser beam through a loaded filter relative to that of a blank filter. The relationship between the optical attenuation and the black carbon content can be written as: .

$$[C_{\text{black}}] = (1/K) \times \text{ATN} , \quad (1)$$

where $\text{ATN} = -100 \ln(I/I_0)$. I and I_0 are the transmitted light intensities for the loaded filter and for the filter blank.

Besides the black carbon, particulate material also contains organic material which is not optically absorbing. The total amount of particulate carbon is then

$$[C_{\text{tot}}] = [C_{\text{black}}] + [C_{\text{org}}] . \quad (2)$$

We define specific attenuation (σ) as the attenuation per unit mass of total carbon:

$$\sigma \equiv \frac{\text{ATN}}{[C_{\text{tot}}]} = K \times [C_{\text{black}}]/[C_{\text{tot}}] . \quad (3)$$

The determination of specific attenuation therefore gives an estimate of black carbon as a fraction of total carbon.

The proportionality constant K , which is equal to the specific attenuation of black carbon alone, was recently shown to have an average value of 20.⁵ In principle the percentage of soot (i.e., primary carbonaceous material) in ambient particles can be determined from the ratio of ambient specific attenuation and an average specific attenuation of major primary sources⁴:

$$[\text{Soot}]/C = \sigma_{\text{ambient}}/\sigma_{\text{source}} . \quad (4)$$

Table I lists the average and extreme values of specific attenuation and the

black carbon fraction of a number of source samples.

The percentage of soot in ambient carbonaceous particulates can be estimated by comparing the σ of sources with that of ambient samples. The fraction of soot is given in Eq. 4. Table II lists the mean specific attenuation of ambient samples (weekends excluded) in order of decreasing σ and soot fractions obtained by using Eq. 4 and $\sigma_{\text{source}} = 5.85$.

Based on this estimate, the New York City carbonaceous aerosol is essentially primary soot. A different value of σ_{source} would certainly change the estimated soot percentage. However, New York City's average soot content would nevertheless remain the highest, irrespective of the actual numerical value of σ_{source} . It is logical that samples from this location have the highest soot content because the site represents a heavily traveled street canyon. Fremont and Anaheim samples have the smallest soot content on the average, as might be expected, because both sites represent receptor sites.

These results demonstrate that soot is certainly a major fraction of ambient particulate carbon at all locations studied. These findings also suggest that in the atmosphere there is a catalytically active material which is present in high concentrations so that the assessment of its role in heterogeneous atmospheric chemistry is warranted.

Soot-Catalyzed SO₂ Oxidation

In this section we review our laboratory results on heterogeneous oxidation on soot particles in air and present results of numerical calculations which suggest that soot-catalyzed oxidation can be an important mechanism for sulfate formation in the atmosphere.

Novakov et al.^{2,8} used photoelectron spectroscopy (ESCA) to study the oxidation of SO₂ on soot particles produced by a propane flame. They found that under some conditions, a significant amount of sulfate can be produced

by the catalytic action of soot particles. Although these early experiments were qualitative, it was nevertheless possible to conclude the following:

1. The reaction product is in a 6^+ oxidation state (i.e., sulfate).
2. Soot-catalyzed oxidation of SO_2 is more efficient at a higher humidity.
3. The oxygen in air plays an important role in SO_2 oxidation.
4. Soot-catalyzed oxidation exhibits a saturation effect.
5. SO_2 can be oxidized on other types of graphitic carbonaceous particles, such as ground graphite particles and activated carbon.

Results from the experiments with combustion-produced soot particles are essentially similar to those obtained for activated carbon by Davtyan and Tkach⁹ and Siedlewski.¹⁰

Soot-catalyzed SO_2 oxidation can proceed by two mechanisms: a "dry" mechanism, in the presence of water, and a "wet" mechanism, when the soot particles are covered by a liquid water layer. The experiments mentioned above⁸⁻¹⁰ involved the dry mechanism. The wet mechanism is much more efficient than the dry and is applicable to situations in plumes, clouds, fogs, and the ambient atmosphere when the aerosol particles are covered with a liquid water layer. The dry mechanism is expected to operate in stacks or under conditions of low relative humidity.

A description of the dry mechanism was given by Yamamoto et al.,¹¹ who studied the reaction kinetics on dry activated carbon in the presence of O_2 and H_2O vapor. The rate of reaction was found to be first order with respect to SO_2 , provided that the concentration of SO_2 was less than 0.01%, and depended on the square root of the concentration of O_2 and H_2O vapor. The activation energy was found to vary from -4 to -7 kcal/mole between 70°C and 150°C , depending on the origin of the activated carbon. Initially the reaction occurs on the surface of both micropores and macropores, and the rate is

constant for a given activated carbon until the amount of accumulated H_2SO_4 reaches about 10% by weight of the carbon. Beyond that amount, the rate gradually decreases with the reaction time until the micropore volume is filled up by H_2SO_4 . The reaction continues only on the macropores at a constant, but much slower, rate.

According to Yamamoto et al.,¹¹ a rate expression (until the amount of H_2SO_4 formed reaches 10% by weight of the carbon) for activated carbon used can be written as follows:

$$\frac{d[\text{H}_2\text{SO}_4]}{dt} = [\text{C}_c] [\text{SO}_2] [\text{O}_2]^{0.5} [\text{H}_2\text{O}]^{0.5} (k_{\text{micro}} + k_{\text{macro}}) e^{-E_a/RT},$$

where t is time $[\text{C}_c]$ is the concentration of carbon, k_{micro} and k_{macro} are the rate constants on the surface of the micropores and macropores, E_a is the activation energy, R is the universal gas constant, and T is absolute temperature.

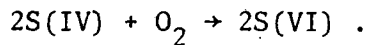
The dry mechanism is relatively inefficient because the reaction product remains on the carbon surface and acts as the catalyst poison. The situation is entirely different when soot (or another carbon) is covered with a layer of liquid water and the catalytic oxidation occurs at the solid-liquid interface: there is constant regeneration of active sites because the reaction product is soluble in water and therefore leaves the soot surface.

Such reactions were studied in detail by Chang et al.¹² and Brodzinsky et al.,¹³ who used both combustion soots and activated carbons. These studies used suspensions of activated carbon in water to which different concentrations of sulfurous acid were added. The results of these studies can be summarized as follows:

1. The reaction rate is first order and 0.69th order with respect to the concentration of carbon and dissolved oxygen respectively.

2. The reaction rate is effectively pH independent (pH < 7.6).
3. The activation energy of the reaction is 11.7 kcal/mole.
4. There is a mass balance between the consumption of sulfurous acid and the production of sulfuric acid.
5. The reaction rate has a complex dependence on the concentration of H_2SO_3 , ranging between a second and zeroth order reaction.

The oxidation of S(IV) to S(VI) can be expressed simply by the symbolic net reaction,



(For this and following reactions, let C_x = carbon surface; S(IV) = $\text{H}_2\text{O}\cdot\text{SO}_2$, HSO_3^- , and $\text{SO}_3^{=}$; S(VI) = HSO_4^- , and $\text{SO}_4^{=}$. The experimental results yield the following empirical rate law for this reaction:

$$\frac{d[\text{S(VI)}]}{dt} = k[C_x][\text{O}_2]^{0.69} \frac{\alpha[\text{S(IV)}]^2}{1 + \beta[\text{S(IV)}] + \alpha[\text{S(IV)}]^2} , \quad (5)$$

where $k = 1.69 \times 10^{-5} \text{ moles} \cdot 31 \cdot \ell \cdot 69 / \text{g} \cdot \text{sec}$,

$\alpha = 1.50 \times 10^{12} \ell^2 / \text{mole}^2$,

$\beta = 3.06 \times 10^6 \ell / \text{mole}$,

$[C_x]$ = grams of carbon/ ℓ ,

$[\text{O}_2]$ = moles of dissolved oxygen/ ℓ , and

$[\text{S(IV)}]$ = total moles of S(IV)/ ℓ .

Using the Arrhenius equation, the rate constant may be expressed as

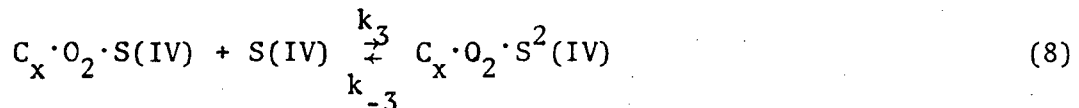
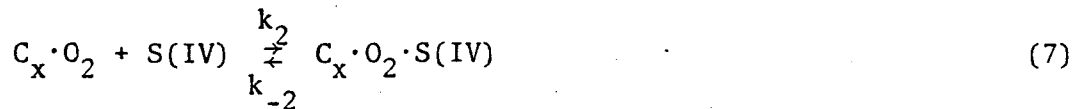
$$k = A e^{-E_a/RT} ,$$

where $E_a = 11.7 \text{ kcal/mole}$, and

$$A = 9.04 \times 10^3 \text{ moles} \cdot 31 \cdot \ell \cdot 69 / \text{g} \cdot \text{sec}.$$

The reaction rate's being first order with respect to the activated carbon catalyst is representative of a surface catalysis. The reaction will then

proceed via the adsorption of the reaction species onto a catalytically active site. A series of adsorption steps can explain the fractional and varying order of reaction with respect to O_2 and $S(IV)$ and are proposed here in the following four-step reaction:



Benner et al.¹⁴ have extended the above research by laboratory fog chamber studies on dispersed water droplets that contain soot particles. In these experiments particles of Nuchar-SN, pre-extracted to remove SO_4 , were resuspended by nebulization; and the resultant mist was passed over heated water and subsequently cooled to cause the nebulized particles to grow into larger droplets (Fig. 1). These droplets were exposed to SO_2 and introduced into the bottom of the fog chamber. Particulate samples were collected at the top of the chamber and analyzed for $SO_4^{=}$ by liquid chromatography and for carbon by combustion.

When pure water droplets were exposed to SO_2 in the fog chamber, the rate of SO_4 formation ($\mu g SO_4/m^3 \cdot h$) equals $4.06 (\text{ppm } SO_2)^{.0007}$ (Fig. 2). If the droplets contained Nuchar-SN particles, the rate of SO_4 formation was found to be significantly faster than for pure water droplets. For example, wet soot particles exposed to 0.007 ppm SO_2 produced SO_4 faster than pure water droplets

exposed to 222 ppm SO_2 . The data for SO_4 formation by wet soot particles are plotted with open circles in Fig. 2. When the soot-droplet data in Fig. 2 are normalized to $[C]$, the carbon-normalized rate of SO_4 formation can be plotted as shown in Fig. 3 (open circles). The equation which best fits the carbon-normalized soot droplet data is $\text{SO}_4^= / C \cdot h = 0.222(\text{SO}_2)^{.147}$.

In Fig. 3 a family of curves shows the rate of SO_4 formation, normalized to C , as a function of $[\text{SO}_2]$ for various initial $[\text{H}^+]$'s. This family of curves was calculated from the rate expression in Eq. 5. The pH effect is related to the absorption of SO_2 by the droplet. The droplet with the lower initial pH will have a lower equilibrium $[\text{S(IV)}]$, and the oxidation rate will be similarly slower. The family of curves was calculated assuming that the droplet had a specified initial pH and equilibrated with SO_2 and 320 ppm CO_2 to produce a certain equilibrium $[\text{S(IV)}]$ before the oxidation started.

The dashed line in Fig. 3 shows the S(IV) oxidation rate expected for wet soot particles in equilibrium with SO_2 . The difference between the fog chamber data and this dashed line is due to the alkaline nature of Nuchar-SN. Neutralization increased the oxidation rate because it brought more $\text{SO}_2 \cdot \text{H}_2\text{O}$ and HSO_3 into the droplet.

Chang et al.¹⁵ have carried out box-type calculations to compare the relative importance of sulfate production mechanisms by soot particles and other mechanisms involving liquid water. The following initial conditions were used in the calculation: liquid water, 0.05 g/m^3 ; SO_2 , 0.01 ppm; O_3 , 0.05 ppm; NH_3 , 5 ppb; and CO_2 , 0.000311 atm. Concentrations of particulate Fe and Mn of 250 ng/m^3 and 20 ng/m^3 respectively were assumed. However, only 0.13% of the total iron and 0.25% of the Mn are water soluble, according to Gordon 35 al.¹⁶ The concentration of soot was taken as $10 \text{ } \mu\text{g/m}^3$.

The results of this calculation, shown in Fig. 4, indicate that O_3 and

soot can be important mechanisms for sulfate aerosol formation. In general the O_3 mechanism is more important under high pH and/or photoactivity conditions when the concentration of O_3 is high, whereas the soot process is more important when the lifetime of fog or clouds is long and the pH of the droplets is low.

Acknowledgment

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Table I. Specific attenuation (σ) and black carbon (BC)(% of total C) of source samples.

Source	# samples	Average		Highest		Lowest	
		σ	% BC	σ	% BC	σ	% BC
Parking garage	12	5.4	27%	7.7	39%	2.25	11%
Diesel	6	5.6	28%	5.7	29%	3.5	18%
Scooter	9	5.1	26%	6.1	31%	4.2	21%
Tunnel	63	6.3	32%	12.5	63%	3.7	19%
Natural gas	6	2.6	13%	3.3	17%	1.9	10%
Garage and tunnel		5.85	29%				

Table II. Mean specific attenuation of ambient samples.

Site	# samples	$\bar{\sigma}$	SDEV	Soot (%)
New York	211	5.69	1.34	97
Gaithersburg	155	4.72	1.51	81
Argonne	221	4.35	1.64	74
Berkeley	513	4.28	1.47	73
Anaheim	444	3.99	1.71	68
Fremont	461	3.74	1.25	64
Denver	42	3.47	1.49	59

Figure Captions

Figure 1. Laboratory fog chamber and associated equipment.

Figure 2. Production of SO_4 in fog droplets.

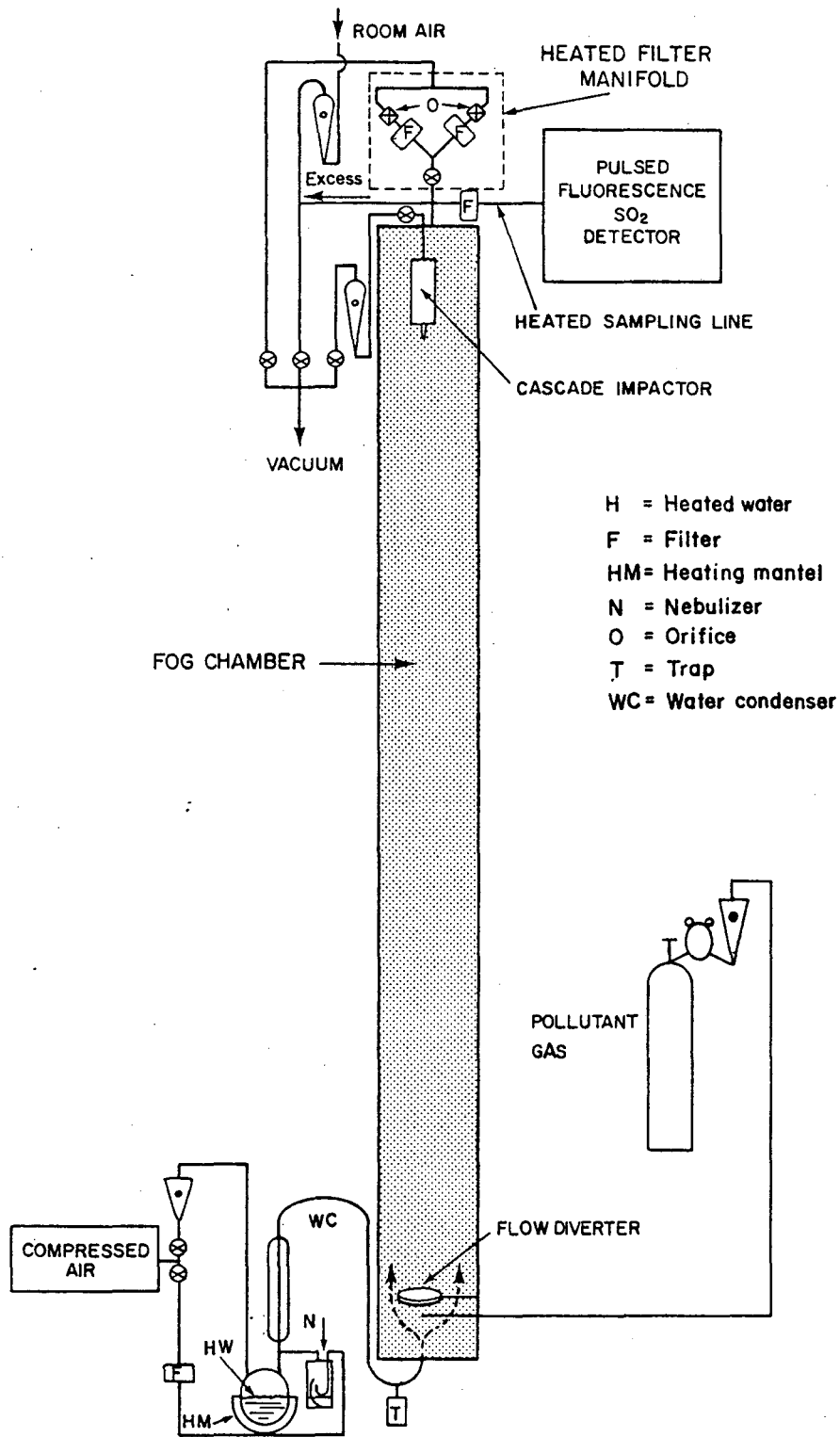
Figure 3. The normalized rate of SO_4 formation in wet soot droplets vs. SO_2 .

The open circles and the solid line drawn through the circles were obtained from fog chamber data. The family of curves labelled $\text{pH}_0 = 2$ through $\text{pH}_0 = 7$ was calculated from the equation

$$\frac{-d[\text{S(IV)}]}{dt} = k[\text{C}] \cdot \frac{\alpha[\text{S(IV)}]^2}{1 + \beta[\text{S(IV)}] + \alpha[\text{S(IV)}]^2}$$

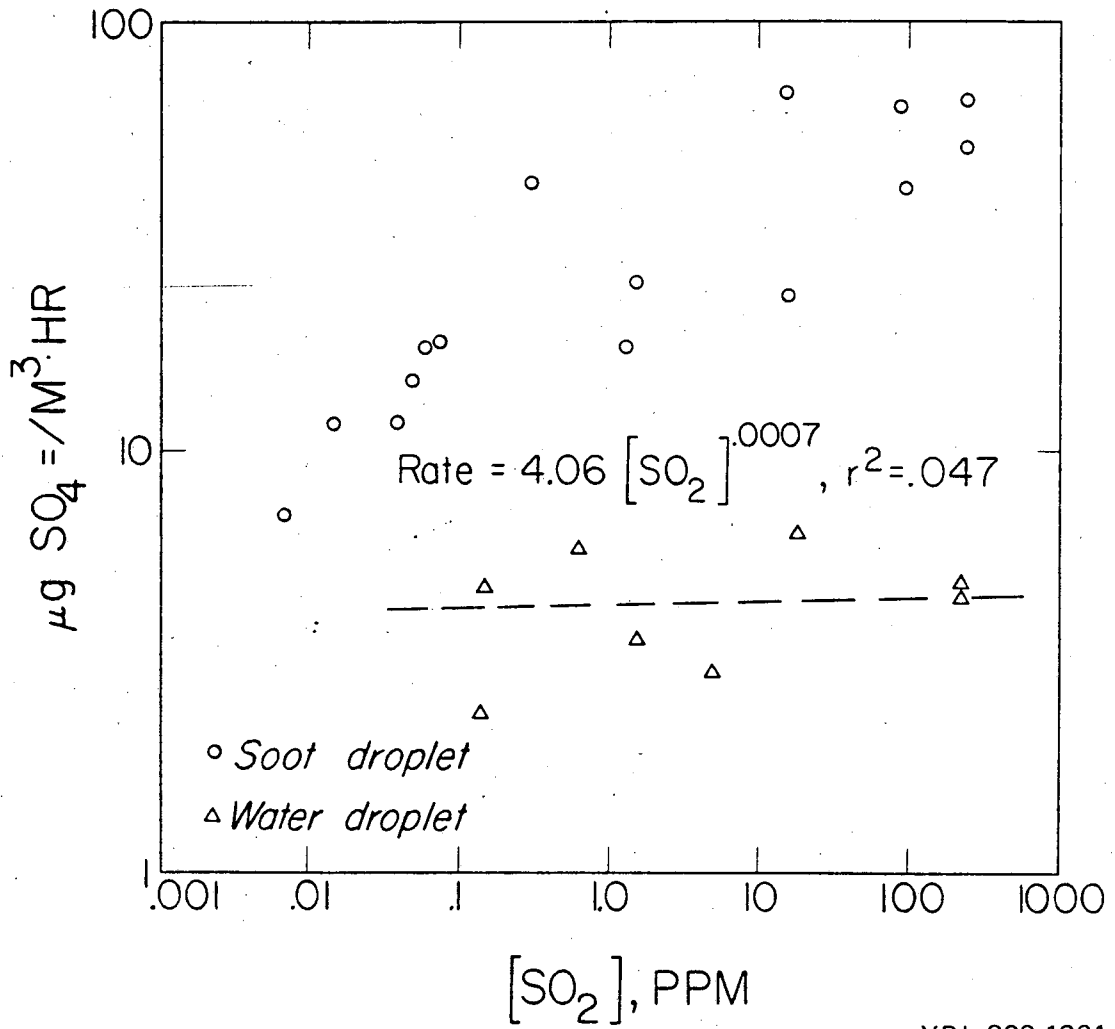
pH_0 is the pH of droplets in equilibrium with SO_2 and CO_2 before the start of the reaction. The dashed line shows the rate for SO_4 formation for wet soot droplets whose pH before exposure to SO_2 and 320 ppm CO_2 is determined by SO_2 - CO_2 - H_2O equilibria. The dashed line is juxtaposed on pH coordinates created by the family of curves labelled $\text{pH}_0 = 2$ through $\text{pH}_0 = 7$.

Figure 4. Comparison of the relative significance of various SO_2 conversion processes in aqueous droplets.



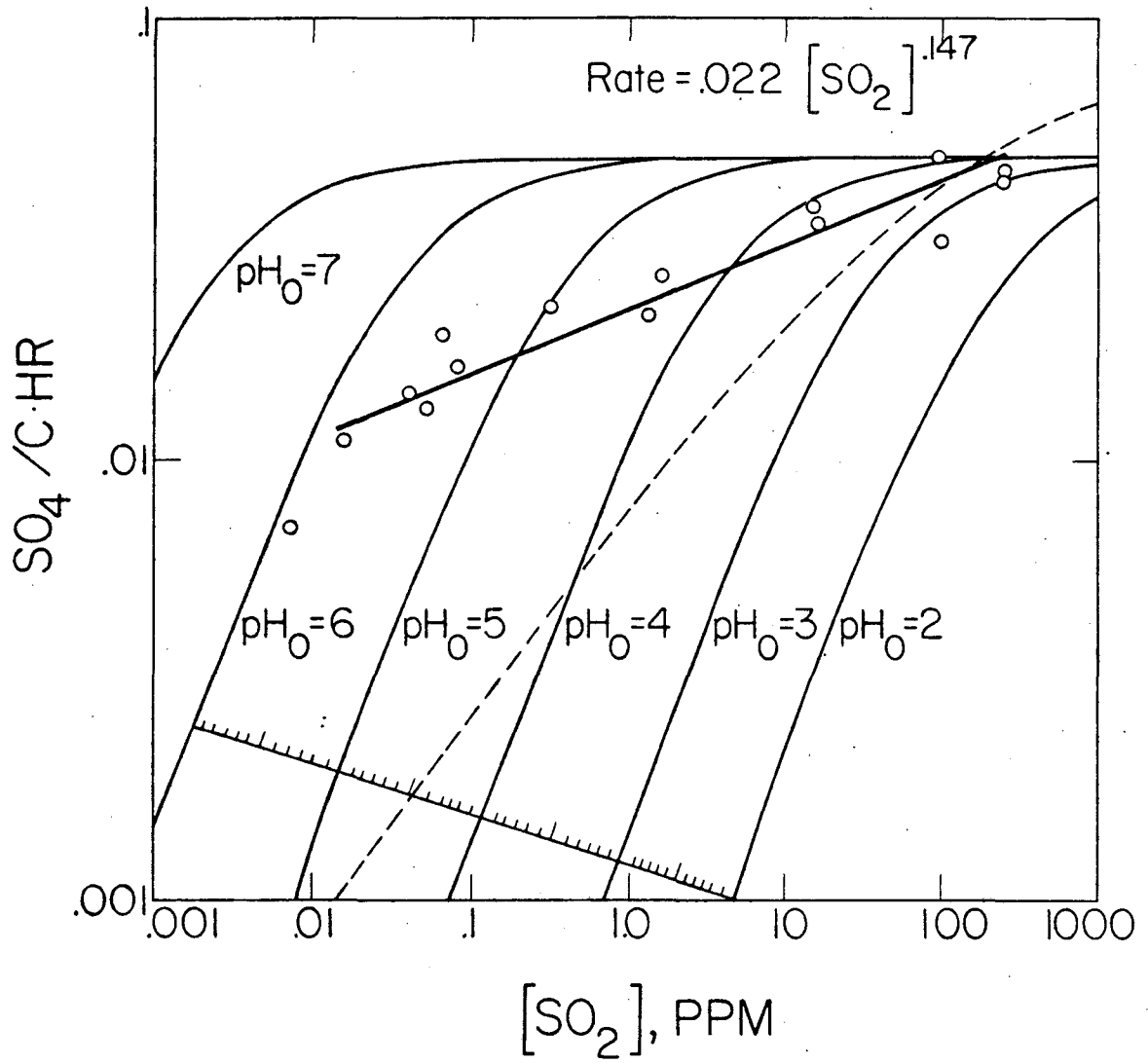
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Figure 1



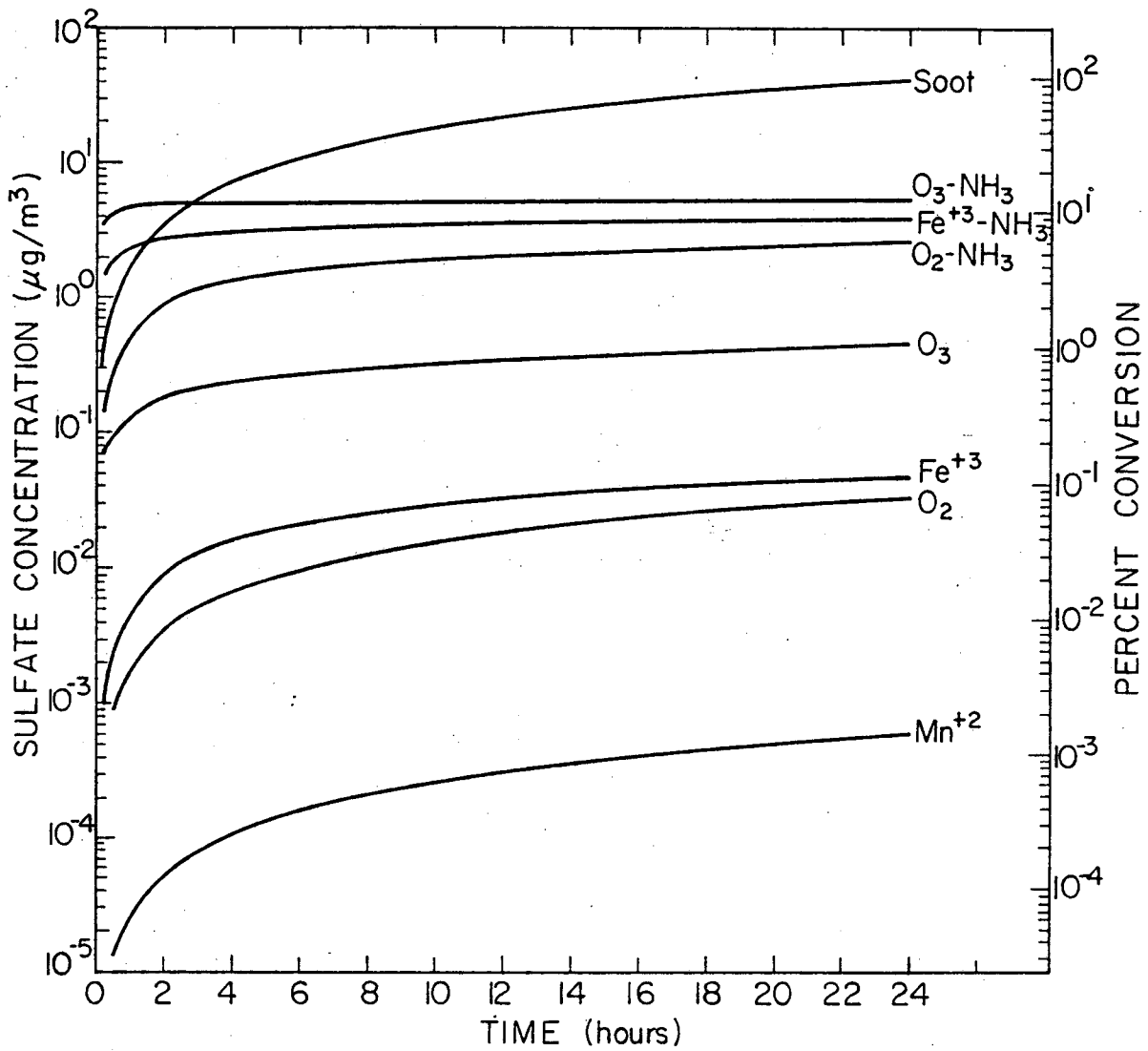
XBL 809-1861

Figure 2



XBL 809-1860

Figure 3



XBL 782-4614

Figure 4

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