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SOOT IN URBAN ATMOSPHERES

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## Lawrence Berkeley Laboratory

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# ENERGY & ENVIRONMENT DIVISION

Presented at the National Research Council Diesel Impacts Study Committee, Los Angeles, CA, May 5-7, 1980

SOOT IN URBAN ATMOSPHERES

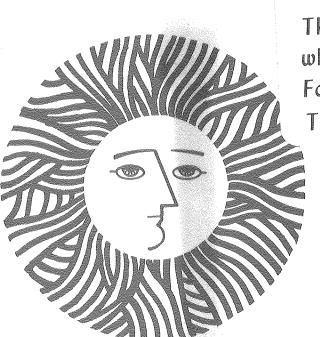
T. Novakov

May 1980



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#### SOOT IN URBAN ATMOSPHERES \*

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Presentation before

NRC, Diesel Impacts Study Committee Environmental Impacts Panel May 5-7, 1980 Los Angeles, California

May 1980

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#### INTRODUCTION

Carbonaceous particles in the atmosphere consist of two major components — graphitic or black carbon (sometimes referred to as elemental or free carbon) and organic material. The latter can either be directly emitted from sources (primary organics) or produced by atmospheric reactions from gaseous precursors (secondary organics). We define soot as the total primary carbonaceous material, i.e., the sum of graphitic carbon and primary organics. In this paper we present data on the concentrations of particulate carbon in a number of locations in the United States and estimate the contribution of soot to the carbonaceous aerosol in urban atmospheres.

The complex set of questions concerning the origin and the chemical and physical characterization of carbonaceous particulates has been central to the program of the Atmospheric Aerosol Research group at Lawrence Berkeley Laboratory (LBL) since the group's beginning in 1972. As a result of our research, we advanced the hypothesis that much of the carbonaceous material in urban environments is soot (primary material). The preliminary results on the origin and nature of carbonaceous particles were first reported at the First Annual NSF Trace Contaminants Conference at Oak Ridge National Laboratory in August 1973, 1 just one year after the start of our research. Additional results strengthening this hypothesis were reported in several publications from 1973 to 1975. 2

#### SAMPLING AND EXPERIMENTAL METHODS

Because relatively few consistent studies of ambient carbonaceous particles have been conducted, in 1977 we established an ongoing routine sampling program at seven sites across the United States. The data consist of information obtained from 24-hour samples (collected every morning Monday through Friday) and

multi-day samples collected over weekends. For the purpose of data analysis, these two data sets can be separated. Table I lists the routine sampling sites with the beginning date of sampling.

Table I. LBL aerosol sampling sites.

Site	Location	Date of first sample
Lawrence Berkeley Laboratory	Berkeley, California	1 June 1977
BAAQMD monitoring station	Fremont, California	15 July 1977
SCAQMD monitoring station	Anaheim, California	19 August 1977
Argonne National Laboratory	Argonne, Illinois	22 January 1979
DOE Environmental Measurements Laboratory	Manhattan, New York	22 November 1978
National Bureau of Standards	Gaithersburg, Maryland	23 January 1979
Denver Research Institute	Denver, Colorado	15 November 1978

The samples are collected in parallel on prefired quartz fiber and Millipore filter membranes. The flow rates employed are in the range of 0.6-2.2 cubic meters of air per square centimeter of active filter area per 24-hour sampling period. The Millipore filter is used for X-ray fluorescence (XRF) elemental analysis and an optical attenuation technique developed in this laboratory. The latter technique gives a measurement that is proportional to the amount of light-absorbing (black) carbon present on the filter. Other analyses are total carbon determination and temperature-programmed evolved gas (CO<sub>2</sub>) analysis combined with optical attenuation measurement (called thermal analysis).

The LBL optical attenuation method<sup>3</sup> compares the transmission of a 633-nm He-Ne laser beam through a loaded filter relative to that of a blank filter. The loaded filters are placed in the beam with the loaded side toward the laser. After multiple scattering through the filter substrate, the light is collected by an f/l lens and focused on a photomultiplier tube. The data presented in

this paper were obtained from particles collected on Millipore filters, but the optical attenuation measurement can be made with a wide variety of filter media. This technique is based on a principle similar to that of the opal glass method used by Weiss et al. and measures the absorbing rather than the scattering properties of the aerosol. For fixed optical constants, a quantitative relationship between the optical attenuation and the black carbon content can be written as:

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

where 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 strongly optically absorbing. The total amount of particulate carbon is then:

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

A fundamental characterization of a particulate sample can be given by its attenuation per unit mass of total carbon, i.e., its specific attenuation,  $\sigma$ :

$$\sigma = \frac{ATN}{[C_{tot}]} = K \times [C_{black}]/[C_{tot}] .$$
 (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. This value was obtained by determining the optical attenuation of 25 samples for which the absolute concentration of black carbon was known.

The validity of the above procedure for estimating the fraction of black carbon from the specific attenuation value can be illustrated by the following example, involving a direct analysis for total carbon and black carbon by thermal analysis. Thermal analysis is used to obtain total carbon, black carbon, organic carbon, and carbonate carbon. A schematic representation of the thermal analysis apparatus used in our studies is shown in Fig. 1. The main components of this apparatus are a quartz tube and a temperature-programmed furnace. The tube is mounted axially inside the furnace. The particulate sample, collected on a prefired quartz filter, is placed in the quartz tube so its surface is perpendicular to the tube axis. The tube is constantly supplied with pure oxygen. The excess oxygen escapes through an axial opening at the end of the tube, while the remainder of the oxygen (and other gases evolved during analysis) passes through a nondispersive infrared analyzer at a constant flow. In addition to the variable temperature furnace, the apparatus also contains a constant temperature furnace, usually kept at about 850°C. The segment of quartz tube inside the constant temperature furnace is filled with a copper oxide catalyst. The purpose of the catalyst is to ensure that carbon-containing gases evolved from the sample are completely converted to CO2. This is especially important at relatively low temperatures when complete oxidation to  ${\rm CO}_{2}$  does not occur.

The actual measurement consists in monitoring the  $\mathrm{CO}_2$  concentration as a function of the sample temperature. The result is a "thermogram" — a plot of the  $\mathrm{CO}_2$  concentration vs. temperature. The area under the thermogram is proportional to the carbon content of the sample. The carbon content is quantitated by calibrating with a calibration gas ( $\mathrm{CO}_2$  in oxygen) and by measuring the flow rate through the system. This calibration is crosschecked by analyzing samples of known carbon content. The thermograms of ambient and source aerosol samples reveal distinct features in the form of peaks or groups of peaks that correspond

to volatilization, pyrolysis, oxidation, and decomposition of the carbonaceous material.

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To determine which of the thermogram peaks corresponds to black graphitic carbon, the intensity of the light beam produced by a He-Ne laser is monitored by a photomultiplier and displayed by the second pen of the chart recorder, simultaneously with the measurement of the CO<sub>2</sub> concentration. In actual experiments the light penetrating the filter is collected by a quartz light guide and filtered by a narrow band interference filter to eliminate the effect of the glow of the furnaces. An examination of the CO<sub>2</sub> and light intensity traces enables the assignment of the peak or peaks in the thermograms corresponding to the black carbon because they appear concomitantly with the decrease in sample absorptivity.

In Fig. 2, a complete thermogram of an ambient sample is shown. The lower trace represents the  ${\rm CO}_2$  concentration vs. the sample temperature, while the upper curve corresponds to the light intensity of the laser light beam that reaches the detector during the temperature scan. Inspection of the thermogram shows that a sudden change in the light intensity occurs concomitantly with the evolution of the  ${\rm CO}_2$  peak at about 470°C. The light intensity  ${\rm I}_0$ , after the 470°C peak has evolved, corresponds to that of a blank filter. This demonstrates that the light-absorbing species in the sample are combustible and carbonaceous. We refer to these species as black carbon. The carbonate peak evolves at about 600°C; and as carbonate is not light absorbing, it does not change the optical attenuation of the sample. In addition to black carbon and carbonate, the thermogram in Fig. 2 also shows several distinct groups of peaks at temperatures below  $\sim 400$ °C that correspond to various organics.

The thermogram in Fig. 2 was obtained with a 1.46-cm-diameter disc cut out of a sample collected on prefired quartz fiber filters. The temperature

ramp rate was 10°C/minute. The integrated area under the  $CO_2$  trace is proportional to the total carbon concentration. For this sample the total carbon concentration, determined by thermal analysis, was 17.9 µg (C)/cm². The black carbon, determined from the thermogram, composes 14% of the total carbon. This value can be crosschecked by using the optical attenuation and total carbon data. The specific attenuation for this sample determined in a separate measurement is  $\sigma \equiv ATN/C = 3.00$ . The estimated percentage of black carbon (as a percent of total C), determined from measurement of optical attenuation and total carbon only is  $100 \times 3.0/20.0 = 15\%$ . This value is in excellent agreement with the percentage of black carbon determined directly from the  $CO_2$  thermogram.

#### RESULTS AND DISCUSSION

#### Total Carbon and Optical Attenuation

Figure 3 shows the variations of 24-hr total carbon (weekends excluded) at the Fremont, California, site. These data cover the period from July 1977 to January 1980. The 24-hr histogram superimposed on the bar diagram represents the monthly averages.

It is evident from Fig. 3 that there are significant day-to-day variations in total carbon. The maximum and minimum daily concentrations differ by an order of magnitude. The monthly averages are at peak values during the November-December periods of each year. The variations in optical attenuation for the same samples are represented in Fig. 4. The pattern of ATN values resembles that of total carbon and shows similar seasonal variations. The specific attenuation (ATN/C) variations represented in Fig. 5 are much less pronounced and show no clear seasonal variations. Similar features of total C, ATN, and ATN/C are also observed at the Berkeley (Figs. 6-8) and Anaheim sites.

At the New York (Figs. 9-11), Gaithersburg (Figs. 12-14), and Argonne (Figs. 15-17) sites, daily and monthly variations of total C and ATN are much

less pronounced than at the three West Coast sites. The ATN/C values at these sites do not show any systematic seasonal trend.

#### Correlations Between Total Carbon and ATN

Statistical analysis of the data shows that there is a strong correlation (r > 0.85) between optical attenuation and total particulate carbon at every site studied.<sup>8,9</sup> Furthermore, a study of a number of source samples shows that there is also a strong correlation between optical attenuation and total carbon for these samples. The correlations between optical attenuation and total carbon for the three California sites, Argonne, and source samples are shown in Fig. 18(a-e).<sup>9</sup>

Results obtained from ambient samples imply that the fraction of graphitic soot to total particulate carbon is approximately constant under the wide range of conditions occurring at a given site. On specific days, however, there can be large variations in the ratio, reflecting the variations in the relative amounts of organic and black carbon. The least squares fit of the data shows regional differences which are related to the fraction of black carbon due to primary emissions. These differences would suggest an increase in the relative importance of the primary component for samples collected respectively at Berkeley, Fremont, Anaheim, and Argonne.

#### Concentrations of Black Carbon

Determination of specific attenuation,  $\sigma \equiv ATN/C$ , enables a straightforward estimation of black carbon. From relation (3) one can calculate black carbon as a percentage of total carbon, and the concentration of black carbon in  $\mu g/m^3$ . Table II lists the average specific attenuation ( $\sigma$ ) and black carbon (BC) percentages for all samples (including multi-day samples) analyzed to date. In addition to the average values, the highest and lowest values are given. Based on this estimate, on the average 20% of the total carbon is black carbon. This

fraction can on occasion be as high as 56% or as low as 6%. The latter occurs as a rule when total carbon concentrations are low.

Table II. Specific attenuation  $(\sigma)$  and black carbon (BC)(% of total C) from ambient samples.

Site	Dates on file	# samples	Averag σ %	e Hig BC σ	hest % BC	Loi	west % BC
New York	Nov 78 - Apr 80	439	5.44 2	7% 11.1	56%	2.8	14%
Argonne	Jan 79 - Mar 80	438	4.30 2	2% 9.1	46%	1.1	6%
Gaithersburg	Jan 79 - Mar 80	381	4.33 2	2% 8.0	40%	1.8	9%
Denver	Nov 78 - May 79	141	3.23 1	6% 5.7	29%	1.4	7%
Anaheim	Aug 77 - Jan 80	852	3.70 1	9% 9.6	48%	0.8	4%
Fremont	Jul 77 - Mar 80	924	3.55 1	8% 8.3	42%	1.6	8%
Berkeley	Jun 77 - Apr 80	998	4.09 2	0% 9.2	46%	1.2	6%

Table III lists the concentrations of total particulate carbon and estimated concentrations of black carbon in  $\mu g/m^3$  for the same samples as Table II.

Table III. Carbon concentrations  $(\mu g/m^3)$ .

	Aver	erage Highest		est	Lowest	
Site	С	BC	С	BC	С	BC
New York	15.2	4.2	53.1	12.6	3.4	0.6
Argonne	8.1	1.7	25.1	5.2	3.1	0.2
Gaithersburg	6.1	1.4	17.6	5.6	2.3	0.3
Denver	9.8	1.6	30.8	5.3	4.1	0.2
Anaheim	16.6	3.1	112.9	17.4	3.1	0.3
Fremont	12.0	2.1	75.6	9.2	3.4	0.3
Berkeley	6.7	1.3	31.7	5.2	3.0	0.3

#### Concentrations of Soot

Soot contains not only black carbon but also various organic material.

Because the organic soot component does not absorb light, the specific attenuation of soot is much less than 20, the  $\sigma$  value of pure black carbon. Table IV lists the average and extreme values of specific attenuation and the black carbon fraction of a number of source samples.

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

		Average		Highest		Lowest	
Source	# samples	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%				

The percentage of soot in ambient carbonaceous particulates can be estimated by comparing the  $\sigma$  of sources with that of ambient samples. The fraction of soot is given by

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

Table V lists the mean specific attenuation of ambient samples (weekends excluded) in order of decreasing  $\sigma$  and soot fractions obtained by using relation (4) and  $\sigma_{\rm source}$  = 5.85.

Table V. Mean specific attenuation of ambient samples.

Site	# samples	ō	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

Based on this estimation, the New York City carbonaceous aerosol is essentially primary soot. A different value of  $\sigma_{\hbox{source}}$  would certainly change the

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estimated soot percentage. However, New York City's average soot content would nevertheless remain the highest, irrespective of the actual numerical value of  $\sigma_{\rm source}$ . It is logical that samples from this location have the highest soot content because the site is representative of a heavily traveled street canyon. Fremont and Anaheim samples have on the average the smallest soot content, as may be expected, because both sites represent receptor sites. According to the above estimate, Denver has the smallest specific attenuation value. It is possible that high-altitude combustion results in increased emissions of primary organics; however, we note that the number of samples from this location is small compared to that from other sites, so these results should be taken with caution.

It is instructive to present the specific attenuation data in the form of histograms representing their frequency of occurrence. Histograms for New York and Fremont (Fig. 19) show that the occurrence of high specific attenuation samples is much greater for New York than for Fremont. Histograms for other sites are shown in Figs. 20 and 21.

#### Secondary Organics

Results in Table V suggest that the West Coast sites have an organic component that occurs in excess of sources or source-dominated organics. This excess should be equal to the secondary organic material, which can be conveniently identified by the thermal analysis method.

We have already described how thermal analysis can be used to obtain the total carbon, black carbon, organic carbon, and carbonate carbon. The greatest strength of this method, however, is its ability to "fingerprint" source-produced carbonaceous particles and their contribution to the ambient aerosols. As an illustration, in Figs. 22 and 23 we show the thermograms of a sample collected in Manhattan (high  $\sigma$ ) and one collected in Berkeley (low  $\sigma$ . The two thermograms

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are substantially different. Common features of both samples are the black carbon and the group of peaks below  $\sim 250\,^{\circ}\text{C}$ , corresponding to volatile organic compounds. However, the Berkeley sample clearly shows the presence of at least two peaks (marked by asterisks in Fig. 23) which are absent in the thermogram of the New York sample. These peaks are not observed in samples collected in a highway tunnel and a parking garage and can be shown to correspond to secondary species.

Experiments performed in our laboratory with solvent-extracted samples have shown that the volatile organic species (< 250°C) are easily removed with cyclohexane, a nonpolar solvent. This observation, combined with the fact that these low-temperature peaks are seen in source samples, suggests their predominantly primary origin.

#### Secondary Organics and Ozone

It is clear from the results described so far that the ratio of black carbon to total carbon may vary on specific days. However, no large systematic differences are found as a function of the ozone concentration, which is viewed as an indicator of the photochemical activity. This is graphically demonstrated in Fig. 24, which shows the distribution of the ratios of the optical attenuation to total carbon content for ambient samples from all the California sites taken together, subdivided according to peak hour ozone concentration. Clearly there is no trend for high-ozone days to be characterized by aerosols which have a significantly reduced black carbon fraction. This places a low limit on the importance of secondary organic particulates formed in ocrrelation with the ozone concentration.

#### **ACKNOWLEDGMENT**

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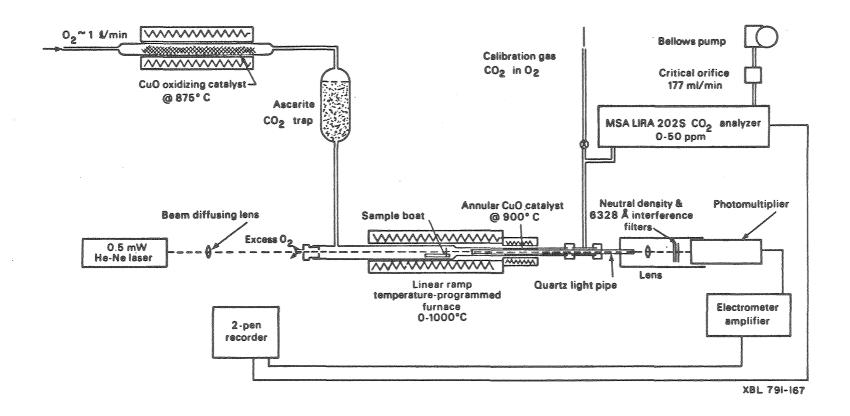


Figure 1.

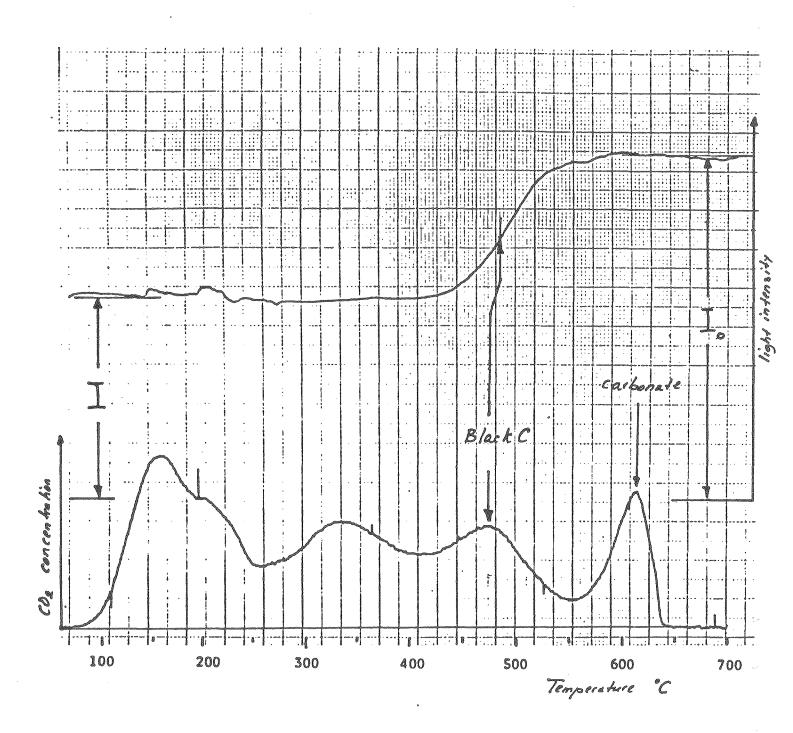


Figure 2.

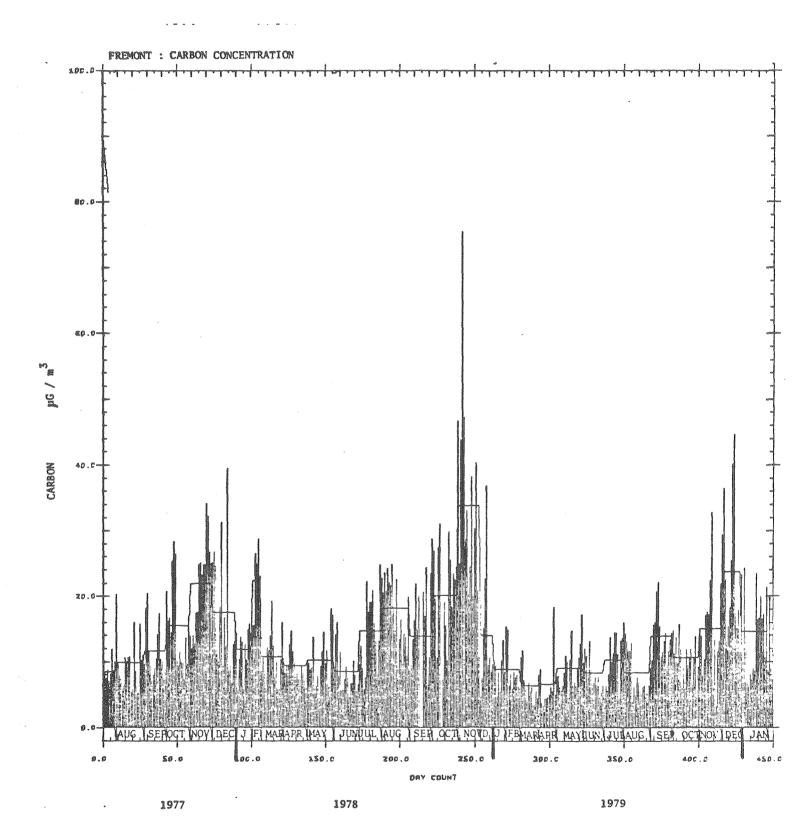


Figure 3.

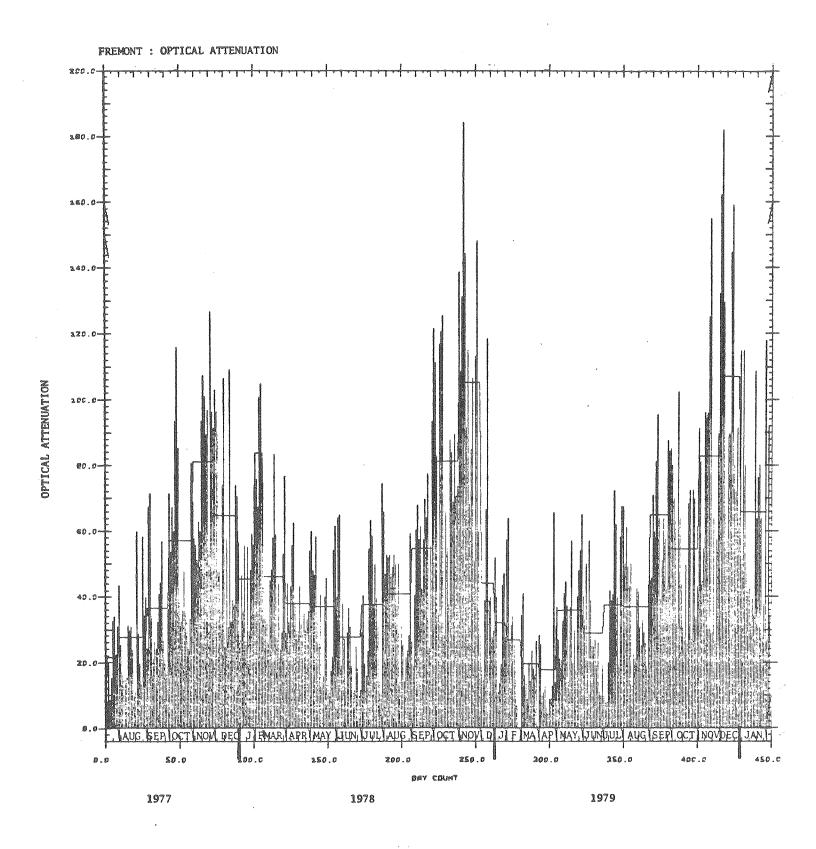


Figure 4.

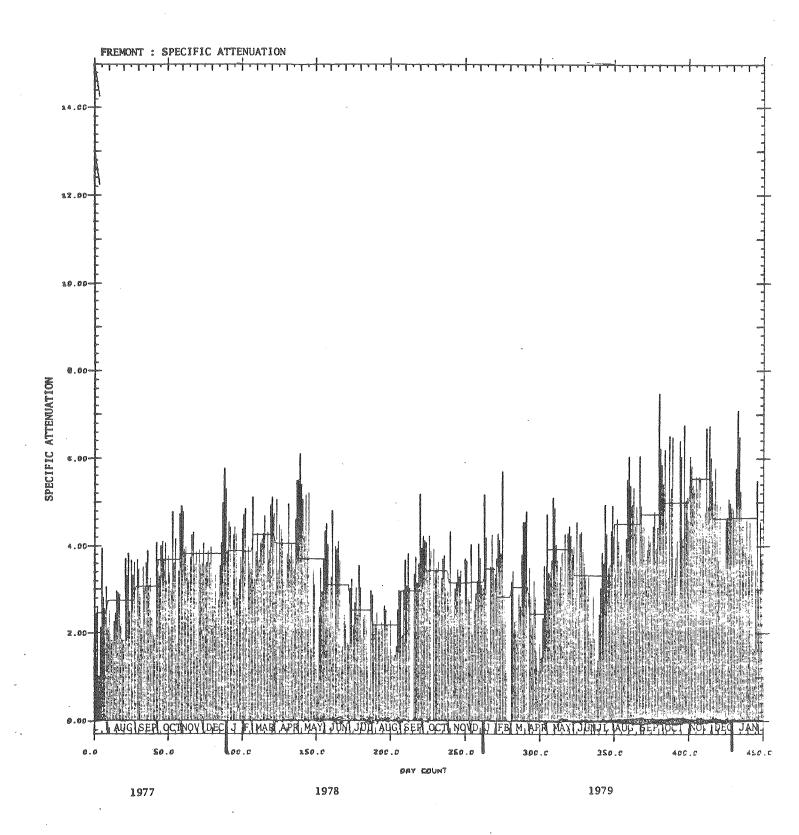
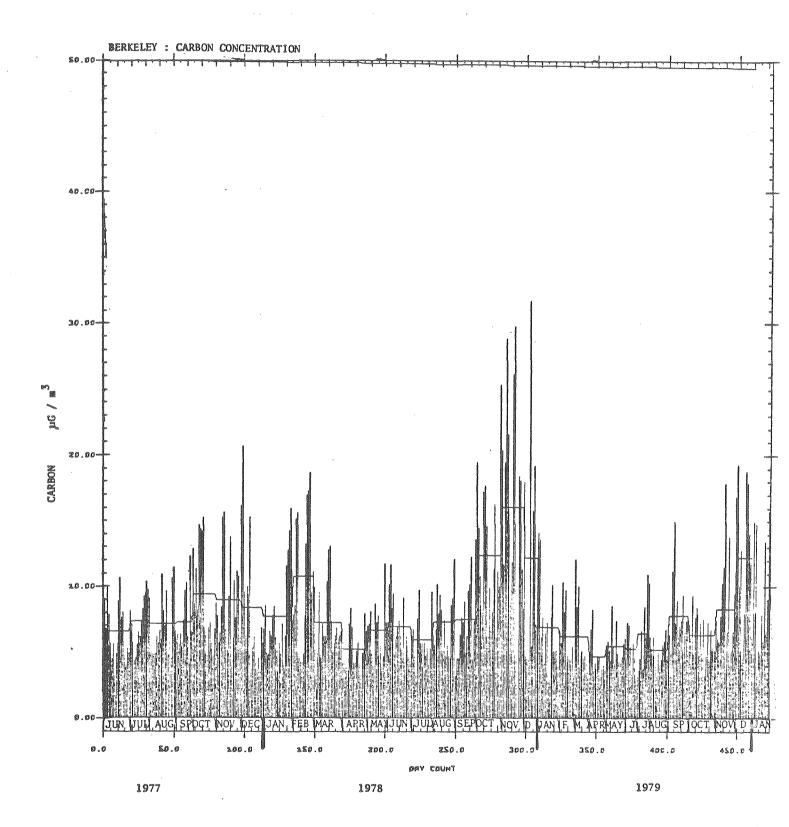


Figure 5.



Fiure 6.

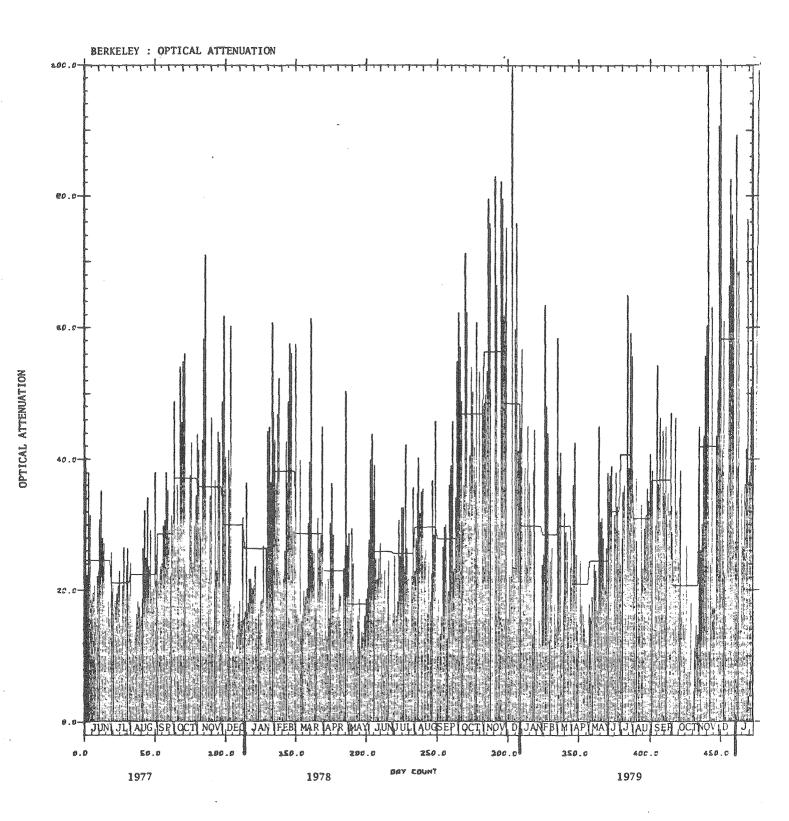


Figure 7.

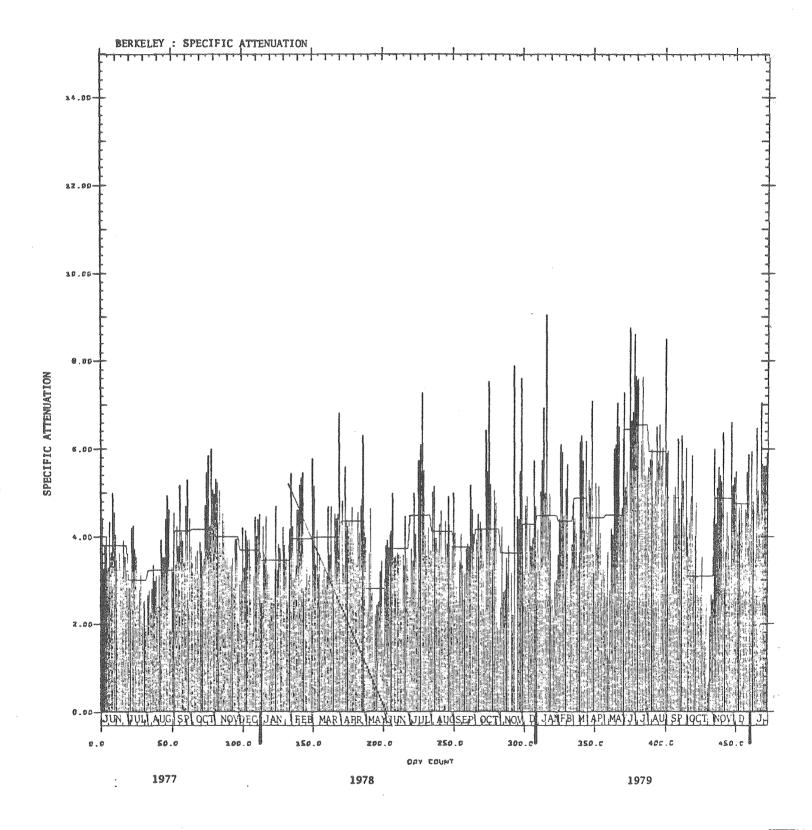


Figure 8.

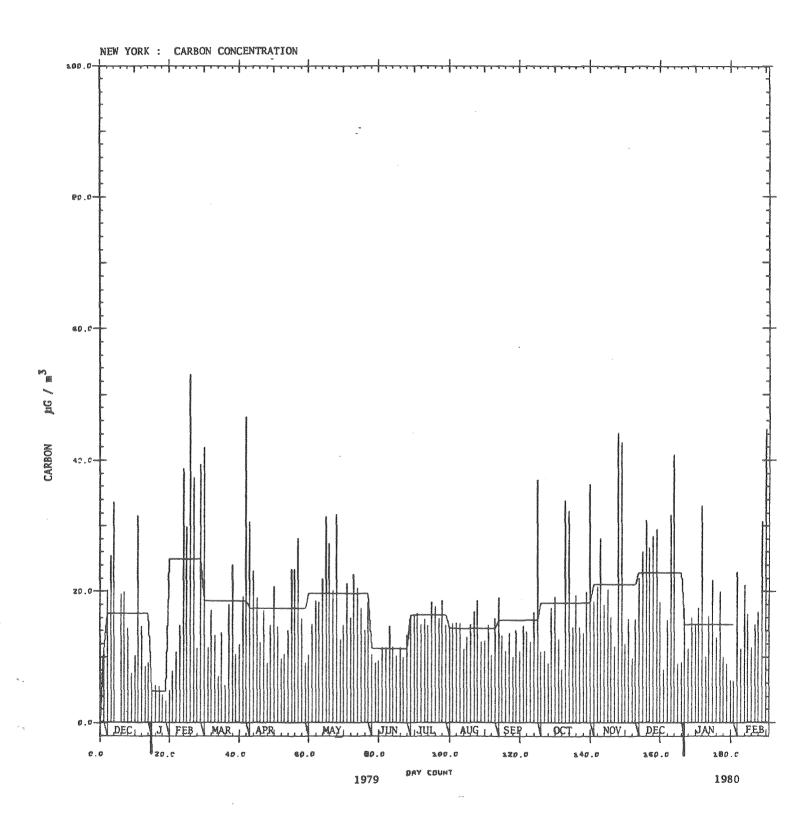


Figure 9.

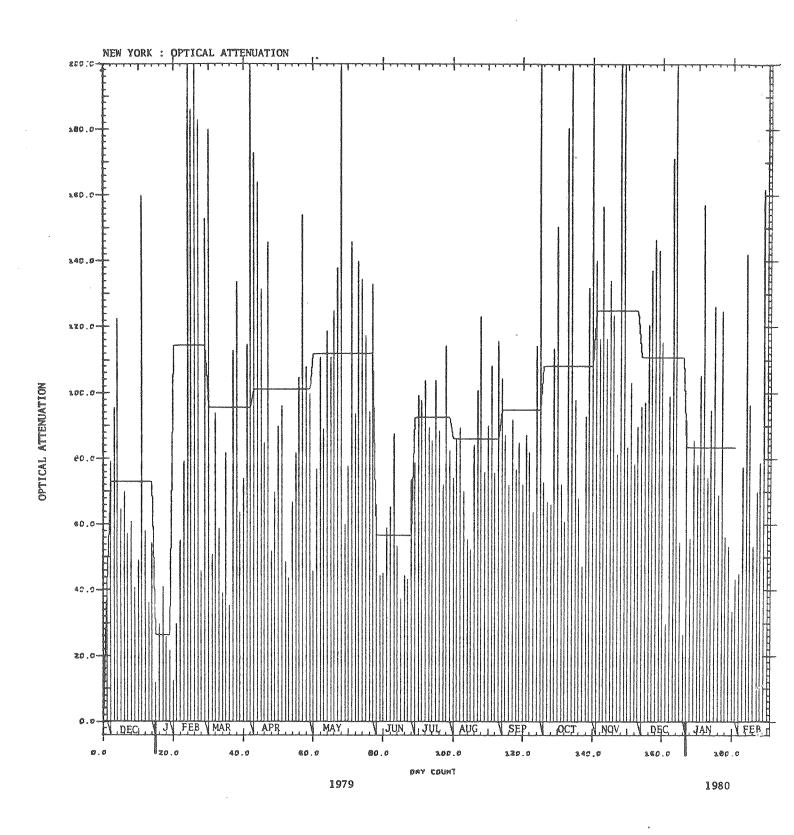


Figure 10.

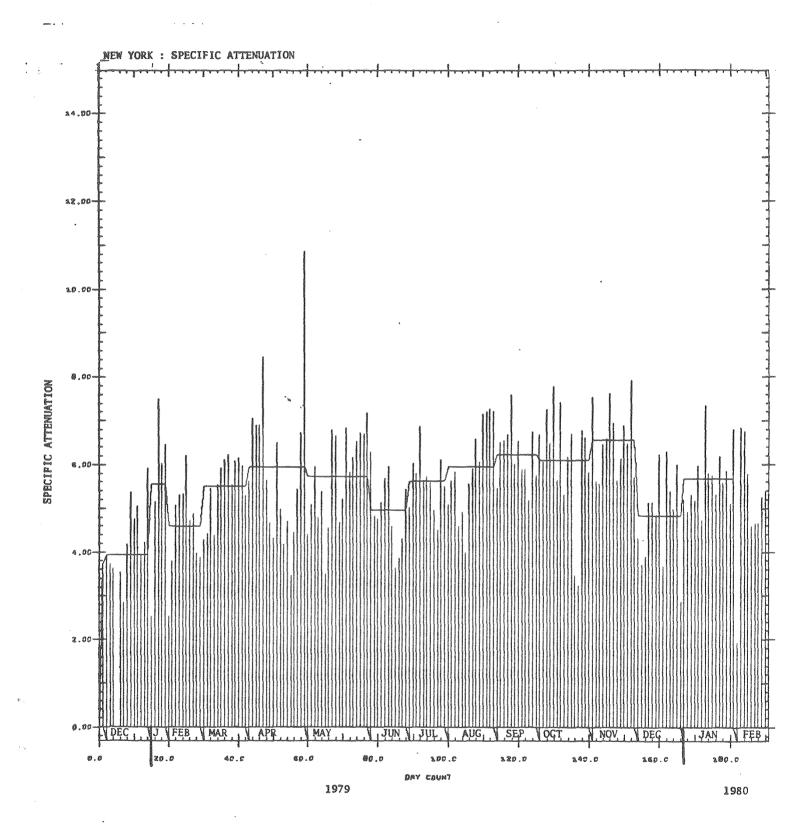


Figure 11.

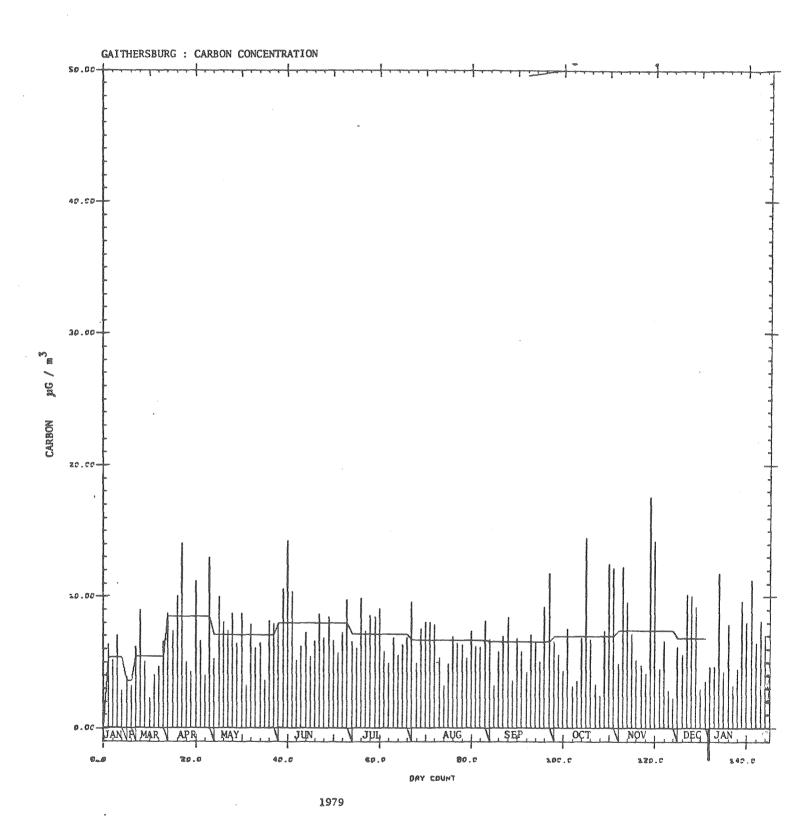


Figure 12.

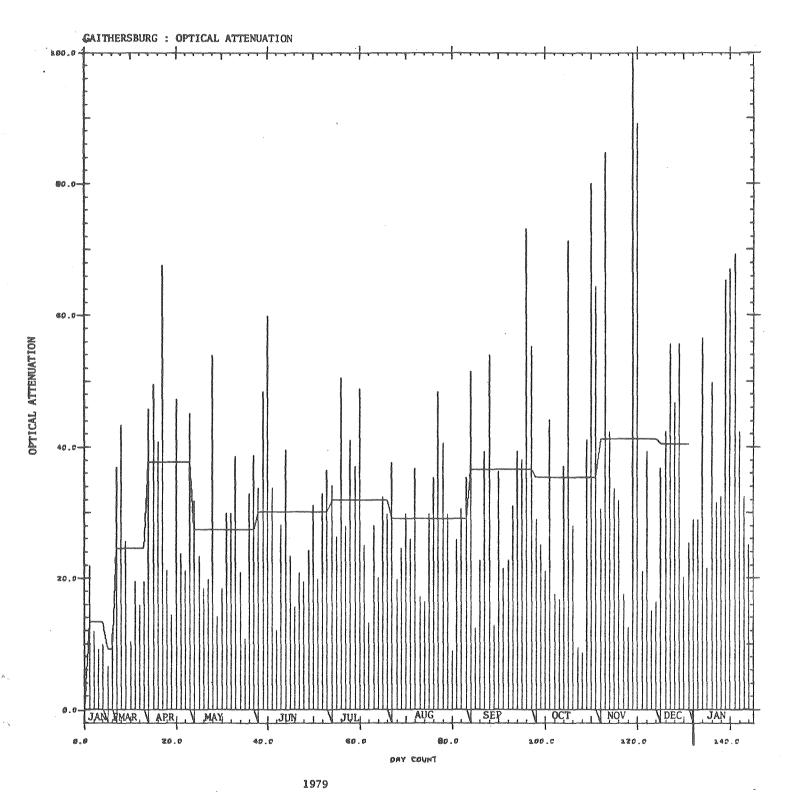
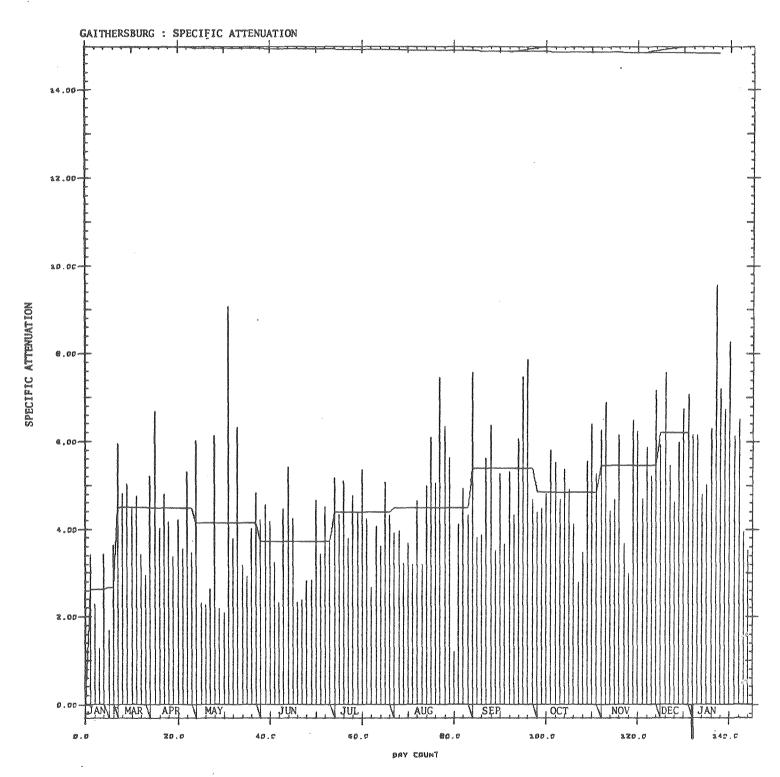


Figure 13.



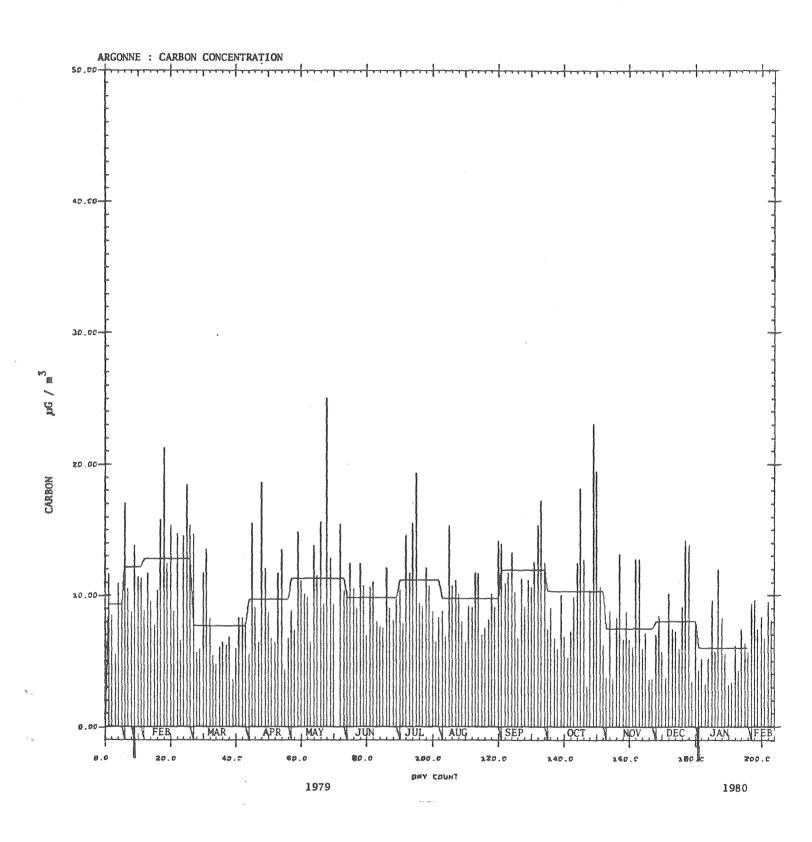


Figure 15.

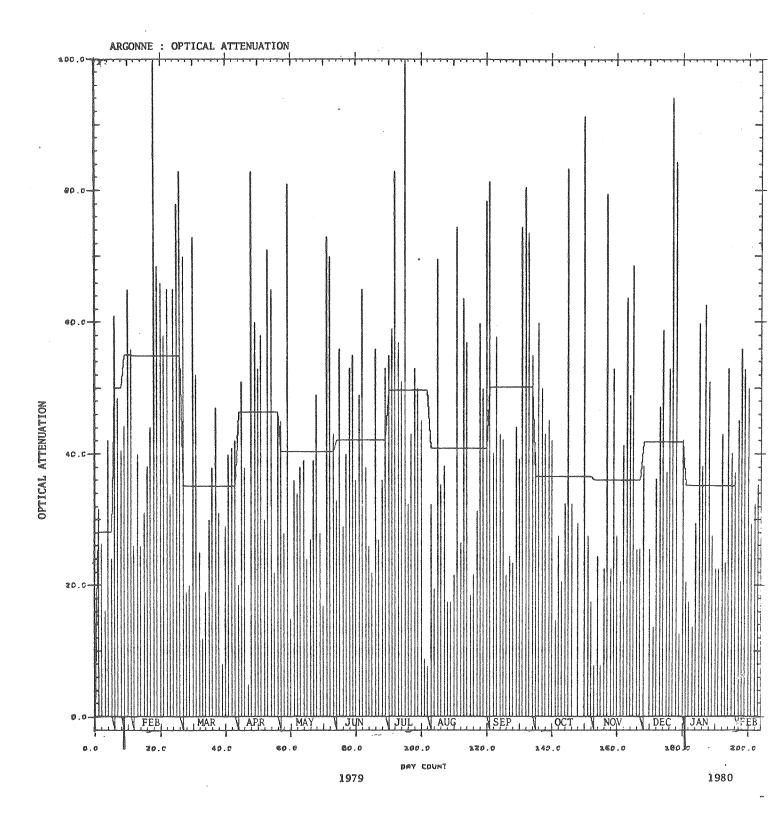


Figure 16.

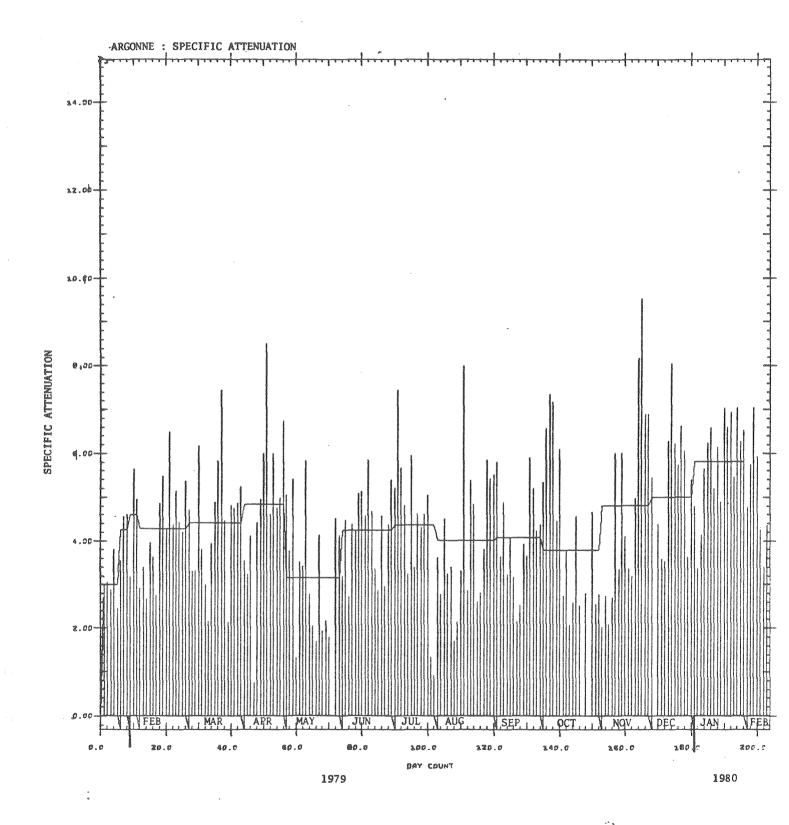


Figure 17.

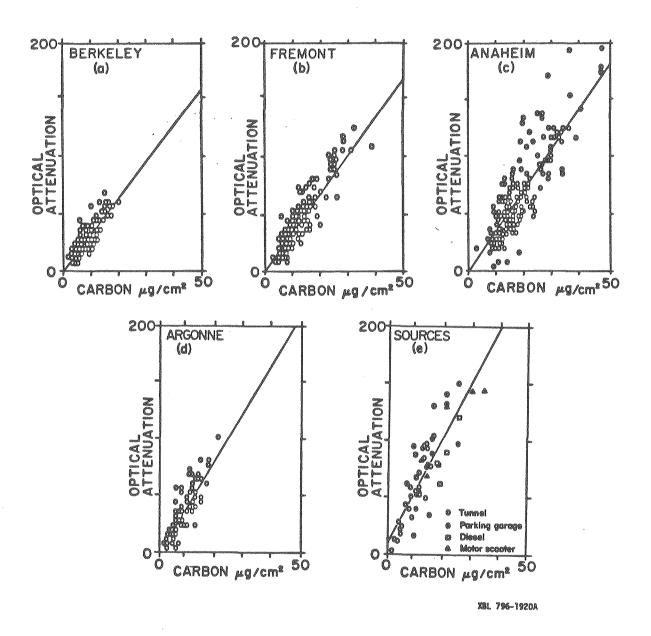


Figure 18.

```
HISTOGRAM OF ATN/ C FOR SITES NYC
FOR DATES 27 NOV 78 TO 14 APR 80
NPOINTS = 211
         MEAN = 5.69
                   SDEV = 1.34 NO WKENDS
  18 %
                      X
                      X o
                     XXX .X
                   XXXXXX XX
                  XXXXXXX XX
                  *XXXXXXX XXX
               XX XXXXXXXXXXXXXXXX
               4.00
         2.00
                     6.00
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   XX XXXXXXXXXXX.
 10.00
     4.00
         6.00
             8.00
 2.00
```

HISTOGRAM OF ATN/ C FOR SITES FRE FOR DATES 18 JUL 77 TO 12 PAR 80 NPOINTS = 461 MEAN = 3.74 SDEV = 1.25 NO WKENDS

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HISTOGRAM OF ATM/ C FOR SITES AND
FOR DATES 22 AUG 77 TO 29 JAN 80
NPOINTS = 444 MEAN = 3.99 SDEV = 1.71 NO WKENDS
  34 8
           · X XX
          XXXX.XXXX
          XXXXXXXXX
         X.XXXXXXXXXXXXX
         2.00 4.00 6.00
                      8.00 10.00
                                ATNI
```

HISTOGRAM OF ATN/ C FOR SITES BRK

FOR DATES 1 JUN 77 TO 17 APR 80

NPOINTS = 513 MEAN = 4.28 SDEV = 1.47 NO WKENDS

```
HISTOGRAM OF ATN/ C FOR SITES DNV
FOR DATES 16 NOV 75 TO 22 MAY 79
NPOINTS = 42 MEAN = 3.47 SDEV = 1.49 NO WKENDS
  10 %
                XX
            XX X
           ******
      XX X
      2.00 4.00 6.00 8.00 19.00
                                       ATN/
HISTOGRAM OF ATN/ C FOR SITES CH!
FOR DATES 23 MAR 78 TO 27 MAR 80
NPOINTS = 221 MEAN = 4.35 SDEV = 1.64 NO WKENDS
  15 %
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          X . X
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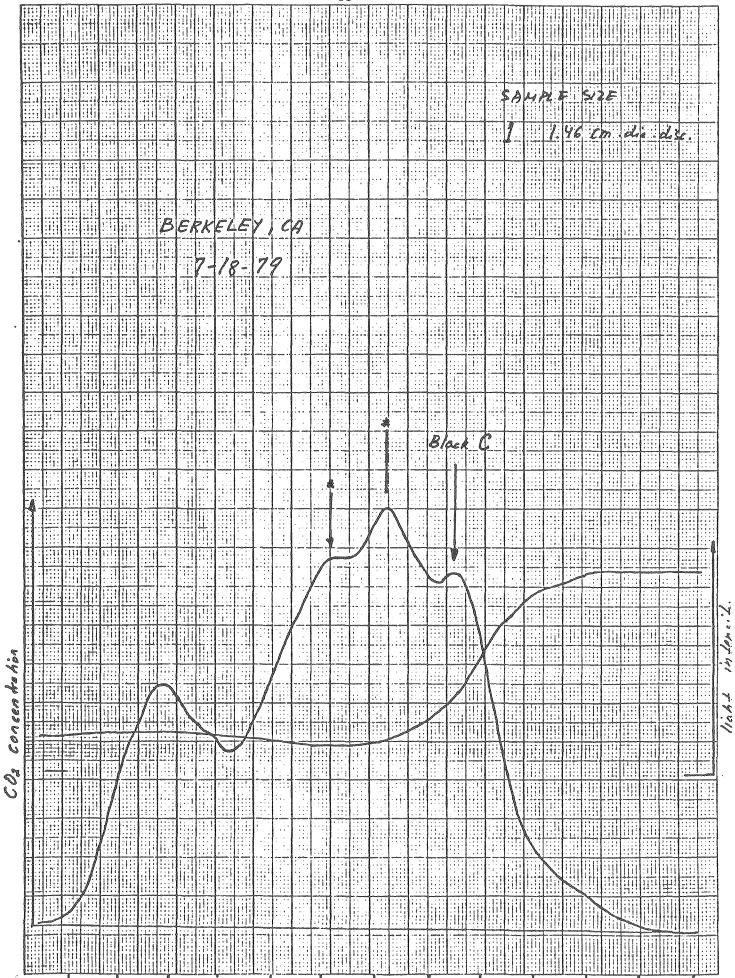
Figure 21.

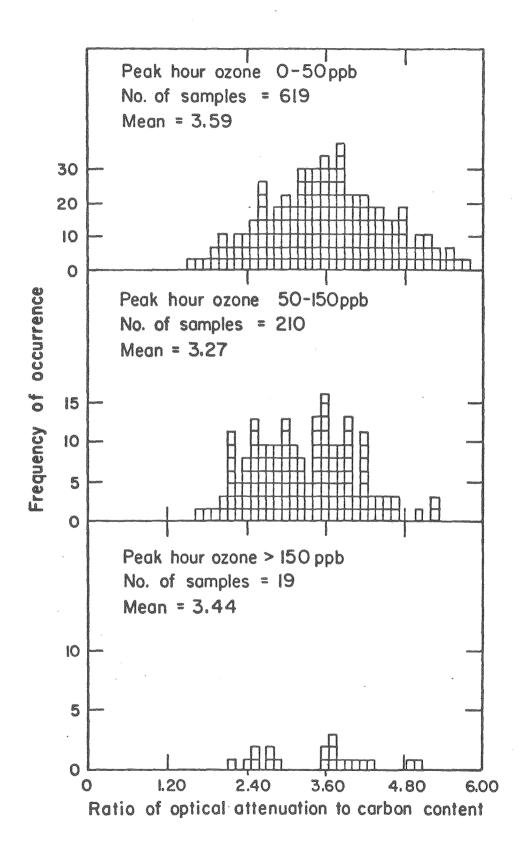
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