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Analysis of Carbon Particle Deposition on Teflon Filters using Fourier Transform Infrared Spectroscopy

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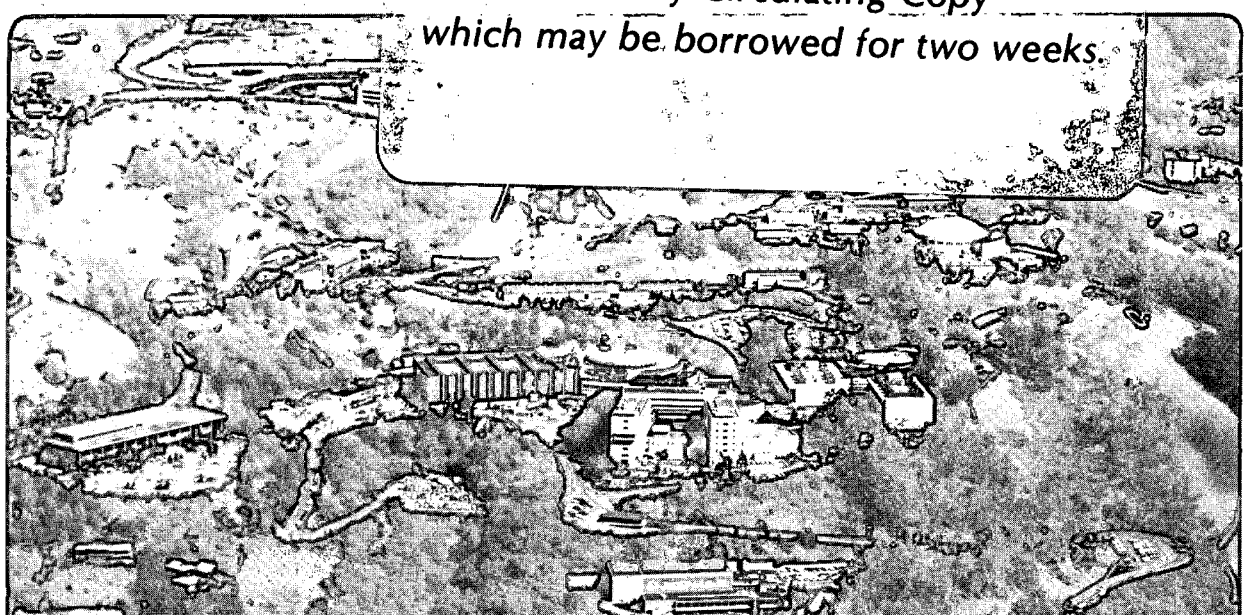

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ANALYSIS OF CARBON PARTICLE DEPOSITION ON TEFLON FILTERS USING FOURIER TRANSFORM INFRARED SPECTROSCOPY

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ABSTRACT

Fourier transform infrared absorption measurements of particulate elemental carbon are compared with results obtained using a thermal combustion method of analysis. Ambient air samples were collected on Teflon* and quartz filters on each day during the sampling study. The quartz filters were analyzed for elemental and organic carbon using a thermal combustion method of analysis. The Teflon* filters were analyzed for elemental carbon by infrared transmission in the region 650 - 666 cm^{-1}. Good correlation was found between the infrared absorbance on the Teflon* filters and the elemental carbon determined from analysis of the quartz filters. Calibration methods for the infrared technique are discussed.

INTRODUCTION

Carbonaceous particles, along with ammonium sulfate and ammonium nitrate, constitute the primary particulate species in the fine fraction (less than 2.5 μm aerodynamic diameter) of ambient aerosols. Particles in this size range scatter visible light very efficiently contributing to visibility

degradation. In addition, carbon particles are the primary light absorbing species in ambient aerosols and they play an important role in the radiative heat balance of the atmosphere. They may also pose a long term health hazard (Daisey, 1980). Carbonaceous particles are a complex mixture of many different compounds but they can be generally divided into elemental and organic fractions each of which can be associated with both anthropogenic and natural sources (Novakov, 1984). Elemental carbon is produced solely in combustion processes. We will present the results of an air sampling study in which two methods of analysis for elemental carbon particles, collected concurrently on quartz and Teflon* filters, were compared.

A variety of analytical methods are used to determine the concentration of organic and elemental carbon collected on air filters. The most widely used procedures for carbon aerosol analysis are thermal methods that determine both volatile (organic) and non-volatile (elemental) carbon (Huntzinger et al., 1982; Cadle, 1982) collected on quartz filters. Organic carbonaceous compounds can also be analyzed with solvent extraction methods (Grosjean, 1975). Non-extractable carbon is assumed to be elemental carbon. Both thermal and solvent extraction methods destroy the filter sample during the analysis. Simple, rapid, and non-destructive optical techniques can be used to measure elemental carbon independently of organic carbon (Lin, 1973; Rosen, 1983). These techniques assume that the absorption of light is due predominantly to elemental carbon since it can be shown that carbon particles absorb much more visible light than any other major aerosol species present in the ambient atmosphere.

Recently another optical method for particle analysis on Teflon* filters has been reported. McClenny et al. (1985) have shown that ammonium and sulfate can be measured directly on Teflon* filters using fourier transform

infrared (FTIR) transmission spectroscopy. Their results showed the equivalence of X-ray fluorescence and FTIR spectroscopy for the quantitative determination of sulfate. Sulfate measurements made previously (Pollard, 1987) have established the equivalence of the FTIR technique to analysis by ion chromatography for the determination of sulfate. In addition to the discrete absorption peaks associated with the vibrational modes of specific molecular species, the mid-infrared spectrum of these ambient aerosols shows continuous absorption across the entire spectrum which is characteristic of carbonaceous particles. To the extent that a relationship between this absorption and the elemental carbon content can be established, a method for non-destructive automated analysis of carbon particle concentration as well as that for sulfate, ammonium, and nitrate particles could result.

The purpose of this study was to:

1. Develop analytical methods to quantitatively determine elemental carbon particle concentration on Teflon* filters.
2. Establish the equivalence of the FTIR carbon measurement to a thermal method of carbon measurement.
3. Develop automated methods of sample manipulation and analysis for use in large sampling studies.

In pursuit of these goals we participated in the Carbonceous Species Methods Intercomparison (CARB) Study conducted by the California Air Resources Board during August of 1986 in Glendora, CA. The primary purpose of the CARB study was to collect and analyze aerosol carbon species by various methods to establish a methods intercomparison data base.

EXPERIMENTAL SECTION

Environmental Monitoring and Services, Inc. (EMSI) collected ambient air samples at Citrus College in Glendora, California during the period August

12-21, 1986. The samples were collected for the 12-hour intervals, 0800-2000 and 2000-0800 each day. Three air samplers, operating in parallel, collected 108 samples of which 65 quartz filters were analyzed by EMSI using a thermal combustion method (Howes, 1987) and 30 Teflon* filters were analyzed by fourier transform infrared spectroscopy. In addition, five Teflon* filter field blanks were analyzed by FTIR as well. All filters were analyzed at the conclusion of the air sampling study.

The air samplers were modified Sierra/Andersen Model 245* automated dichotomous samplers. The modification consisted of removing the virtual impactors and replacing them with identical inlets. Each inlet was equipped with a cyclone to admit particles $\leq$ 2.5 μm aerodynamic diameter. The regulated flow rates were approximately 24 lpm. Each channel has a rotameter to set and monitor the flow rate and a pneumatic controller to regulate the flow rate to within ± 5% of the set point for a filter pressure drop up to 35 cm Hg. The air filters were either 37 mm diameter, 2 micron pore size Teflon* membrane filters supported on a polyolefin support ring or 37 mm diameter quartz fiber filters. The filters were supported in circular polypropylene filter holders. The collection area of the filters is 6.38 cm^2. The filters, in their filter holders, are placed in a circular sample carousel which is then mounted in the air sampler. During the CARB study the samplers were operated in the manual mode.

After the air samples were collected, each Teflon* filter was analyzed nondestructively with the FTIR spectrometer. The infrared analysis was performed on a Nicolet 5DXB* Fourier Transform Infrared Spectrometer equipped with a deuterated triglycine sulfate (DTGS) detector. The spectra were taken in dry purge air using a 2 cm^{-1} resolution over the spectral range 400 - 4600 cm^{-1} and signal-averaged for 100 scans. Spectra of the

individual blank Teflon* filters were measured before air sampling to be used as background reference spectra. The filter spectra were measured again after air sampling. The ratio of the spectra to the open-beam spectra at the time of measurement was calculated and stored as absorbance data files. The background subtraction procedures are described elsewhere (Pollard, 1987).

The FTIR spectrometer was modified for automatic sample analysis by directing the infrared beam out the side of the spectrometer into a chamber containing the filter samples and the DTGS detector. The sample carousel from the Sierra/Andersen* dichotomous sampler can be mounted directly in the sample changer with no handling of the filters. A pair of stepper motors, under direct program control from the FTIR computer, can rotate and horizontally position the sample carousel to move any filter into the infrared beam for analysis. The infrared beam, incident on the exposed side of the filters, is directed down through the filters in the sample carousel to the detector underneath the carousel.

Elemental carbon standards were prepared by depositing propane soot on Teflon* filters using a dichotomous air sampler. The soot deposition on the filters in the fine fraction (< 2.5 μm) channel of the sampler was determined gravimetrically on a Sartorius model 4503 MP6* microbalance (sensitivity 1 μg). Each filter was weighed five times before and after loading with propane soot. The masses of the soot standards ranged from 12 - 80 μg corresponding to areal densities of 2 - 15 $\mu g/cm^2$.

The quartz filters were analyzed by EMSI using a modified Dorhman* carbon analyzer and a Horiba Model PIR 2000* nondispersive infrared (NDIR) CO_2 detector. A 0.25 inch diameter punch from the exposed area of a quartz filter was placed in the analyzer. Organic compounds were volatilized (but not

oxidized) in helium carrier gas to 600°C and transported downstream to a zone packed with manganese dioxide (MnO_2) and heated to 350°C. Here the gaseous hydrocarbon species are converted to CO_2 which is then detected by the NDIR CO_2 detector. The remaining elemental carbon on the quartz punch is heated to a 750°C in a carrier gas of helium containing 2% oxygen. The evolved CO_2 is detected with the NDIR CO_2 detector.

RESULTS AND DISCUSSION

The mid-infrared (400 - 4600 cm^{-1}) absorption spectra of ambient aerosols includes information useful for the determination of the concentration of soot, sulfate, nitrate, and ammonium collected on Teflon* filters. The infrared spectra of ambient aerosol particles has been reported previously (Cunningham, 1974; McClenny et al., 1985; Pollard, 1986). Figure 1 shows mid infrared absorbance spectra from an aerosol sample obtained during the CARB study on August 14-15, 1986 during the 2000-0800 sampling interval. The dashed line is the spectrum of the blank Teflon* filter before sampling (note that the absorbance scale for this spectra is to the right of the figure). The solid line is the spectrum of the collected aerosol particles after subtraction of the background Teflon* filter spectrum. The functional absorbance bands for sulfate, nitrate, and ammonium have been identified. The infrared spectrum of elemental carbon is predominantly continuous across the entire region shown in Fig. 1. Some weak broad absorbance bands due to surface functional groups have been identified in other studies (Akhter, 1985; Friedel, 1970; Mattson, 1969) of carbon soots.

The organic component of soot consists of a complex mixture of many compounds with overlapping infrared spectra (Boone, 1986). Some of the organic functional group infrared absorbance bands have been identified

(Dangler et al., 1987) for ambient aerosols. In our spectra of aerosols on Teflon* filters collected during the CARB study, distinct aliphatic absorbance bands at 2920 cm^{-1} and 2853 cm^{-1} could be seen in 17 filter spectra. Figure 2 is a spectrum obtained from one of these filters. The aliphatic absorbance bands are superimposed on a broad ammonium band between 2800 - 3400 cm^{-1} (Hertzberg, 1964). Organic absorbance bands in other regions of the spectrum have not been observed. Many of these could be obscured by the ammonium, nitrate, sulfate, and Teflon* absorbance bands in the 1000 - 1550 cm^{-1} region. The spectra in which the aliphatic absorbance bands have been observed are identified in Table 1. The organic bands are most distinct when the relative ammonium concentration is low. This was the situation for the samples collected toward the end of the CARB study. No attempt was made to quantitatively determine aliphatic carbon concentrations on the filters.

The presence of the relatively strong aliphatic absorbance bands suggested that other functional group absorbances may interfere with measurements made for other components in other regions of the infrared spectrum. In order to quantitatively determine elemental carbon, a region of the infrared spectrum where organic compounds do not absorb should be chosen for the elemental carbon analysis. A number of articles have been written describing organic compounds that have been identified in ambient aerosols (Daisey, 1980; Van Vaeck, 1978). Figure 3 is an absorbance map (Aldrich, 1978) of some aliphatic hydrocarbons, carboxcylic acids, and nitrogen containing aromatics found in ambient aerosols. In this study the region 650 - 666 cm^{-1} was chosen to measure elemental carbon because:

1. It is unlikely that organic compounds will absorb significantly in this region of the infrared spectrum.

2. There are no interfering absorbance bands due to Teflon*, sulfate, or nitrate in this region of the spectrum.
3. This region is near the 514 cm^{-1} Teflon* band used as a reference for background subtraction. The absorbance values of the background subtracted spectra will be most valid near this Teflon* reference band.
4. Scattering for 0.1 μm particles, a typical diameter for ambient particles, is insignificant at this wavelength. Mie scattering calculations in our laboratory and elsewhere (Jennings, 1978; Roessler, 1982) have shown that the scattering efficiency for particles less than 1 μm in diameter for wavelengths greater than 11 μm (less than 900 cm^{-1}) is zero.

The 65 quartz filters were analyzed for organic and elemental carbon. The 30 Teflon* filters were analyzed for elemental carbon. The aerosols were collected on these filters using three air samplers (samplers 1, 2, and 3) each of which sampled fine fraction particles in two intake channels (channels A and B). Table 1 shows the results of the analysis of each filter in each channel of each sampler, the sampled air volume in cubic meters, and the date and time of sampling. The sampling times nominally correspond to a 0800-2000 and 2000-0800 sampling schedule. Two sets of FTIR results are shown reflecting the two independent calibrations procedures as discussed below.

We can compare the results of the thermal and optical methods by plotting the average value of each method during each sampling interval. Figure 4 is a plot of the daily average measured area in the 650 - 666 cm^{-1} region of the background corrected infrared spectra of the ambient aerosols versus the average elemental carbon results measured by the thermal method on the quartz filters. It can be seen that the results are highly correlated (r = 0.90,

N = 16) except for the one measurement during sampling period from 2000-0800 hours on 8/13 - 8/14 which has an excessively high integrated absorbance area. This measurement has been considered an outlier for all subsequent linear regression calculations but has been included in the table and figures. This measurement is high relative to the other measurements as if it were contaminated by additional carbon material. However sulfate measurements on this same filter did not show it to be an outlier for sulfate analysis (Pollard, 1987). It was also found to be an outlier relative to carbon measurements made by other participants in the CARB study.

Another way to analyze the data in Table 1 is to compare the results based on paired quartz and Teflon* filters from the same sampler for each sampling interval. There were 23 pairs of filters that can be compared in this manner. This comparison can be considered to be a calibration of the FTIR technique using the results of the carbon thermal analysis as a standard reference method. A linear least squares fit line was calculated for this data (slope = 0.104, intercept = 0.276, N = 23, r = 0.870). The measured FTIR elemental carbon particle concentration during the CARB study using this calibration is shown in the second to last column of Table 1. With this calibration procedure it is not surprising that the FTIR measurements agree with the EMSI thermal analysis. Figure 5 shows the results of LBL and two other participants in the CARB study compared to the carbon measured by EMSI. Hansen's (1984) real time measurements were made by an aethalometer. Appel's measurements were made by laser absorption on quartz filters. There is good agreement for elemental carbon measured by all the participants shown in Fig. 5.

An independent calibration was made using soot standards prepared in the laboratory. Propane soot was deposited on Teflon* filters so that the range of blackening of the filters corresponded to the range observed on the CARB samples. Gravimetric analysis showed that the soot mass loadings on the standards were comparable to ambient soot mass loadings on the ambient sample filters. A linear least squares calibration equation relating measured spectral area to propane soot mass concentration was calculated (slope = 0.0268, intercept = 0.0166, N = 8, r = 0.984). The ambient FTIR measured elemental carbon concentration during the CARB study based on this calibration is shown in last column of Table 1.

The ambient elemental soot concentrations calculated from the calibration computed from the absorbance measurements of the propane soot standards are consistently higher than the results calculated from the calibration computed from the results of the thermal methods indicating that there is much less absorption for a given mass concentration of propane soot than for the same mass concentration due to ambient soot. This can be seen by comparing the FTIR results in the last two columns of Table 1. One possible source of error could be that the carbon standards were too heavy due to adsorbed water on the particles or the presence of organic compounds. Although the absorption bands for neither of these compounds could be detected in the infrared spectra of the carbon standards, they could still contribute to some error in the gravimetric analysis of the standards.

Electron micrographs show significant morphological differences between the propane soot and ambient particles. The propane soot is present as long thin agglomerated filaments whereas ambient particles are present as larger diameter granular particles. These morphological dissimilarities and the

different optical cross sections of these particles may also contribute to the observed differences in the results shown in Table 1.

Another significant difference between the propane soot standards and ambient samples is that ambient samples are a mixture of absorbing (carbon) and nonabsorbing (ammonium sulfate, ammonium nitrate) particles. Previous authors (Ackerman, 1981; Bennett, 1982; Clarke, 1982) have determined that mixtures of carbon and ammonium sulfate have different absorption properties than the pure components alone. In addition, the embedding of small particles deep in the fibrous Teflon* filter would increase absorption due the multiple scattering within the filter. We conclude that there are a number of questions that require further investigation before calibration against a direct carbon standard can succeed. However calibration is feasible using thermal combustion analysis of co-collected samples on quartz filters. The thermal combustion analysis can be related to chemical and gravimetric analysis of carbon standards.

CONCLUSIONS

The primary conclusion resulting from this study is that there is a high correlation between the integrated area of selected portions of the infrared spectrum of ambient aerosols and carbon measured by current thermal methods. The spectral regions selected for integration can be chosen to avoid regions of significant organic absorbance and in fact correlate more highly with measurements of elemental carbon than with organic carbon. It is possible to quantitatively determine carbon concentrations on the filter using a suitable calibration method. Sulfate can simultaneously be measured as well. It should also be possible to quantitatively determine ammonium and nitrate. All

four compounds can be measured simultaneously with this simple, rapid, and non-destructive FTIR technique.

At the present time, calibrations must be made by collecting quartz filter samples in parallel with Teflon* filters and analyzing the quartz filters by thermal methods. An independent calibration based on propane soot standards proved to be unsatisfactory. No definitive reasons could be determined why this method of calibration doesn't agree with thermal methods except to note that the propane soot particles exhibited significant morphological differences relative to ambient particles and that they were not present as a mixture with nonabsorbing particles as is the case for ambient particle samples.

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*Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Department of Energy to the exclusion of others that may be suitable.

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TABLE 1. Results of Carbon Particle Analysis for the CARB Study

Sampling Date	Time	Sampler[a]	Sampled Volume m^3	EMSI[c] Organic Carbon[b]	EMSI[c] Elemental Carbon[b]	LBL Elemental Carbon[d] Thermal Calibration[b]	LBL Elemental Carbon[d] Propane Calibration[b]
	0806-2000	1 A	16.56			7.28	14.94
	0806-2000	1 B	16.24	12.11	6.16		
8/12	0806-2000	2 A	16.56	11.74	7.70		
	0806-2000	2 B	16.56	11.89	7.36		
	0806-2000	3 A	16.56			9.29	17.68
	0806-2000	3 B	16.40			7.76	15.53
	2127-0800	1 B	14.41	5.52	3.10		
8/12	2127-0800	2 A	14.49	6.04	3.74		
8/13	2127-0800	2 B	14.31	5.86	3.10		
	1014-2000	1 B	13.43			3.57	11.21
	1035-2000	2 A	13.00			5.15	14.61
8/13	1035-2000	2 B	13.00	14.05	6.11		
	0932-2000	3 A	14.45			5.47	13.69
	0932-2000	3 B †	14.45			8.02	18.07
	2011-0705	1 B	14.90	7.11	3.81		
8/13	2011-0800	2 A †	16.32			11.39	21.13
8/14	2011-0800	2 B	16.32	5.64	4.05		
	0830-2000	1 A †	16.26			8.17	16.29
	0830-2000	1 B	16.10	15.20	7.25		
8/14	0805-2000	2 A	17.01	14.04	9.58		
	0805-2000	2 B	16.85	14.59	8.72		
	0805-2000	3 A	17.01			9.69	17.79
	2010-0809	1 A	16.85			4.10	9.71
	2010-0809	1 B	16.60	6.27	4.11		
8/14	2010-0809	2 A	17.27	6.02	3.92		
8/15	2010-0809	2 B	16.77	4.35	4.42		
	0825-2000	1 A	6.16			6.78	14.26
	0825-2000	1 B	16.00	12.94	7.58		
8/15	0825-2000	2 A	16.40			6.23	13.21
	0825-2000	2 B	15.68	13.60	7.31		
	0825-2000	3 A	16.08			6.33	13.63
	2014-0800	1 A †	16.27			2.65	7.84
8/15	2014-0800	1 B	16.15	5.06	3.23		
8/16	2014-0800	2 A	16.80			3.36	8.65
	2014-0800	2 B	16.31	4.65	2.43		
	0811-2000	1 A	15.94			4.56	10.99
8/16	0811-2000	1 B	15.86	13.92	6.48		
	0811-2000	2 A	16.39	11.56	6.30		
	0811-2000	2 B	16.02	13.59	6.58		

TABLE 1. continued

Sampling Date	Time	Sampler[a]	Sampled Volume m^3	EMSI[c] Organic Carbon[b]	EMSI[c] Elemental Carbon[b]	LBL Elemental Carbon[d] Thermal Calibration[b]	LBL Elemental Carbon[d] Propane Calibration[b]
8/16	0811-2000	3 A	16.31	11.20	4.22		
	0811-2000	3 B	15.85	11.24	5.50		
	2010-0800	1 A	16.41			3.37	8.88
	2010-0800	1 B	16.25	9.57	4.14		
8/16	2010-0800	2 A	15.93	8.09	4.58		
8/17	2010-0800	2 B	15.69	9.48	3.66		
	2010-0800	3 A	16.65	6.31	2.96		
	2010-0800	3 B	15.77	7.83	2.91		
	0808-2002	1 A †	16.50			3.74	9.38
	0808-2002	1 B	16.34	13.54	3.97		
8/17	0808-2002	2 A	16.02			6.14	13.38
	0808-2002	2 B	15.77	13.95	4.30		
	0808-2002	3 A	16.75	12.74	3.84		
	0808-2002	3 B	15.86	12.76	3.71		
	2009-0800	1 A †	16.33			.71	4.87
8/17	2009-0800	1 B	16.24	6.85	1.94		
	2009-0800	2 A †	16.00			.74	5.02
8/18	2009-0800	2 B	15.61	7.23	2.13		
	2009-0800	3 A	16.49	5.17	2.61		
	2009-0800	3 B	15.92	5.56	2.00		
	0806-2000	1 A †	15.60			5.05	12.00
	0806-2000	1 B	16.39	12.31	4.19		
8/18	0806-2000	2 A	15.75	9.16	4.27		
	0806-2000	2 B	15.35	10.43	2.94		
	0806-2000	3 A	16.23	9.34	4.18		
	0806-2000	3 B	15.12	8.32	3.70		
	2009-0800	1 A	16.14			1.78	6.57
	2009-0800	1 B	16.14	6.35	1.62		
8/18	2009-0800	2 A	15.90	6.68	1.68		
8/19	2009-0800	2 B	15.35	7.51	1.42		
	2009-0800	3 A	14.88	8.00	1.84		
	2009-0800	3 B	14.88	7.56	1.73		
	0812-2000	1 A †	16.07			4.89	11.41
8/19	0812-2000	1 B	16.07	10.74	3.65		
	0812-2000	2 A †	15.83			5.08	11.88
	0812-2000	2 B	15.28	10.54	3.38		
	0812-2000	3 A	14.81	11.00	3.60		
	0812-2000	3 B	14.81	9.08	3.28		
8/19	2010-0800	1 A †	16.15			2.84	8.20
8/20	2010-0800	1 B	16.07	9.12	3.17		

TABLE 1. continued

Sampling Date	Time	Sampler[a]	Sampled Volume m3	EMSI[c] Organic Carbon[b]	EMSI[c] Elemental Carbon[b]	LBL Elemental Carbon[d] Thermal Calibration[b]	LBL Elemental Carbon[d] Propane Calibration[b]
8/19	2010-0800	2 A †	16.31			2.31	7.31
8/20	2010-0800	2 B	16.15	9.09	3.09		
	2010-0800	3 A	14.27	9.70	3.04		
	2010-0800	3 B	14.19	8.80	2.67		
	0808-2000	1 A	16.09			8.62	17.16
	0808-2000	1 B	16.25	12.07	7.50		
8/20	0808-2000	2 A	16.17	12.93	6.59		
	0808-2000	2 B	15.69	12.22	5.60		
	0808-2000	3 A	13.23	13.99	5.50		
	0808-2000	3 B	12.72	12.62	6.05		
	2010-0800	1 A	16.29			4.67	10.92
	2010-0800	1 B	16.13	9.94	4.43		
8/20	2010-0800	2 A	16.13	9.87	5.47		
	2010-0800	2 B	15.81	10.55	4.03		
8/21	2010-0800	3 A	13.72	10.86	3.98		
	2010-0800	3 B	12.83	9.66	3.42		

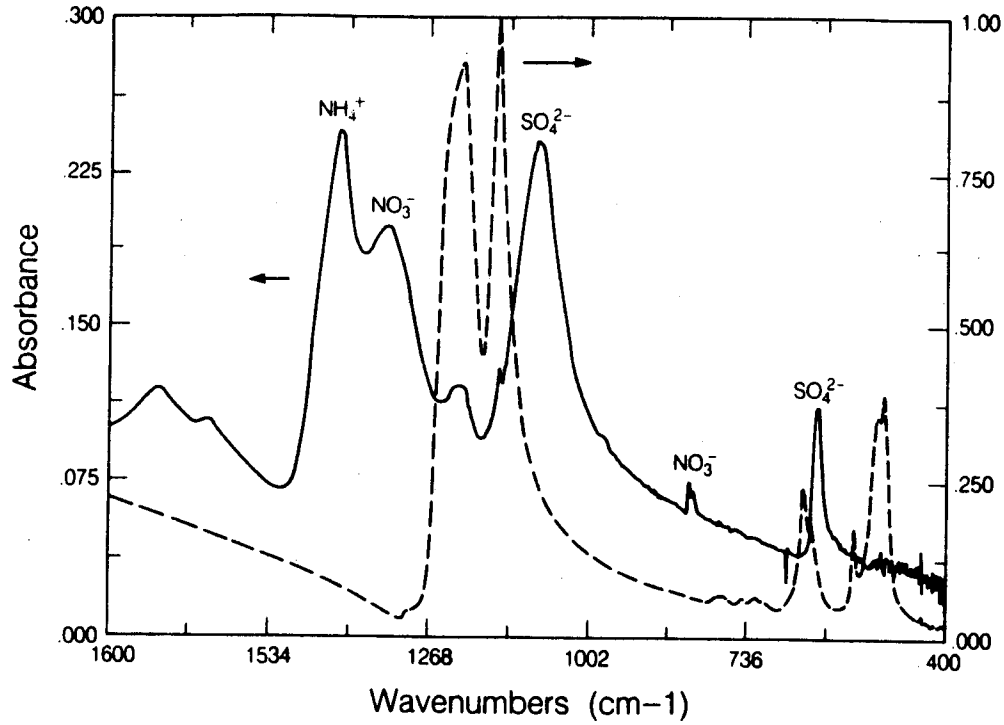
†Distinct aliphatic absorption bands

[a]Three dichotomous samplers were used. The A and B refer to the two separate inlets in each sampler.

[b]All measurements in $\mu g/m^3$.

[c]EMSI organic and elemental carbon on quartz filters by thermal combustion analysis.

[d]LBL measured elemental carbon on Teflon* filters using infrared absorbance.



XBL 879-10344

Figure 1. The background corrected infrared spectrum of an ambient aerosol sample (solid line) and the blank Teflon* filter (dashed line) before sampling. The arrows indicate the respective absorbance scales for the two spectra.

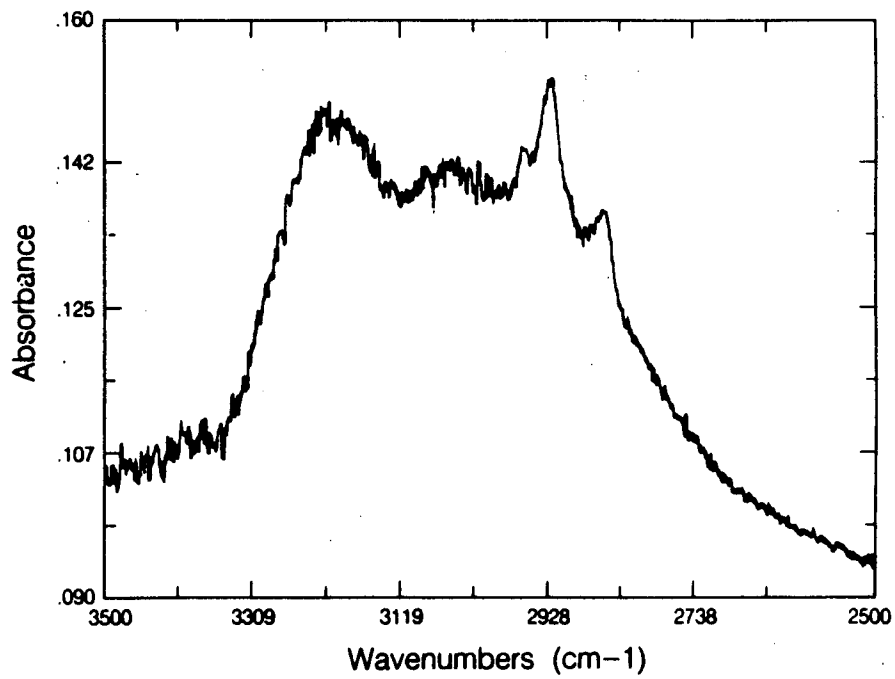


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Figure 2. The infrared spectrum of an ambient aerosol sample showing the aliphatic absorption bands at 2920 cm^{-1} and 2853 cm^{-1} superimposed on a broad ammonium absorption band.

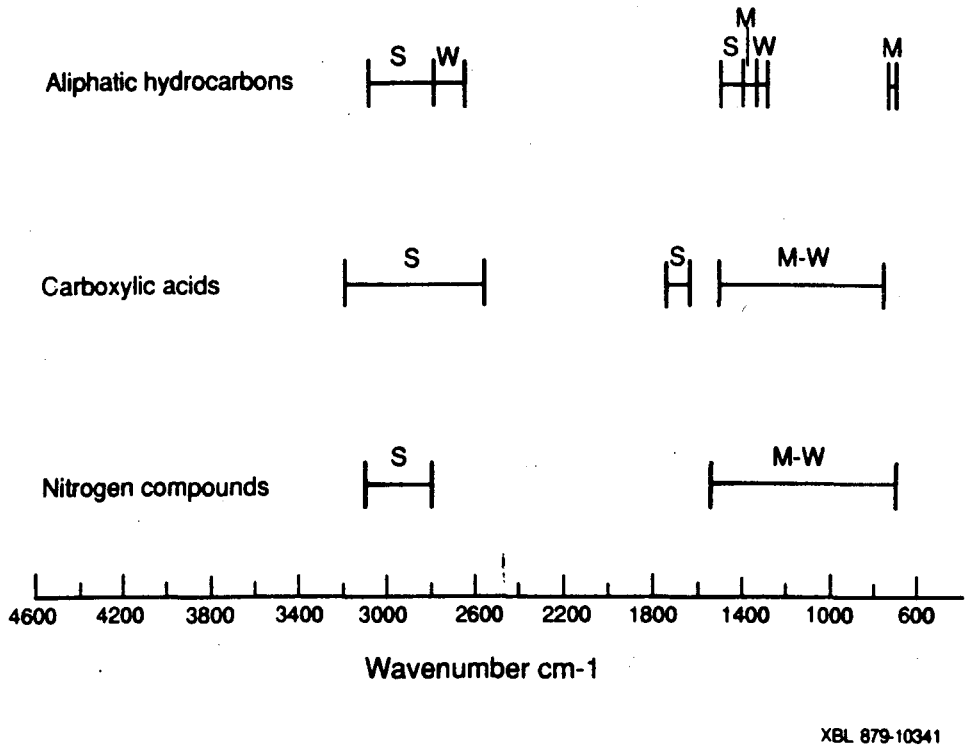

Figure 3. Regions of absorption due to some organic compounds found in ambient aerosols.

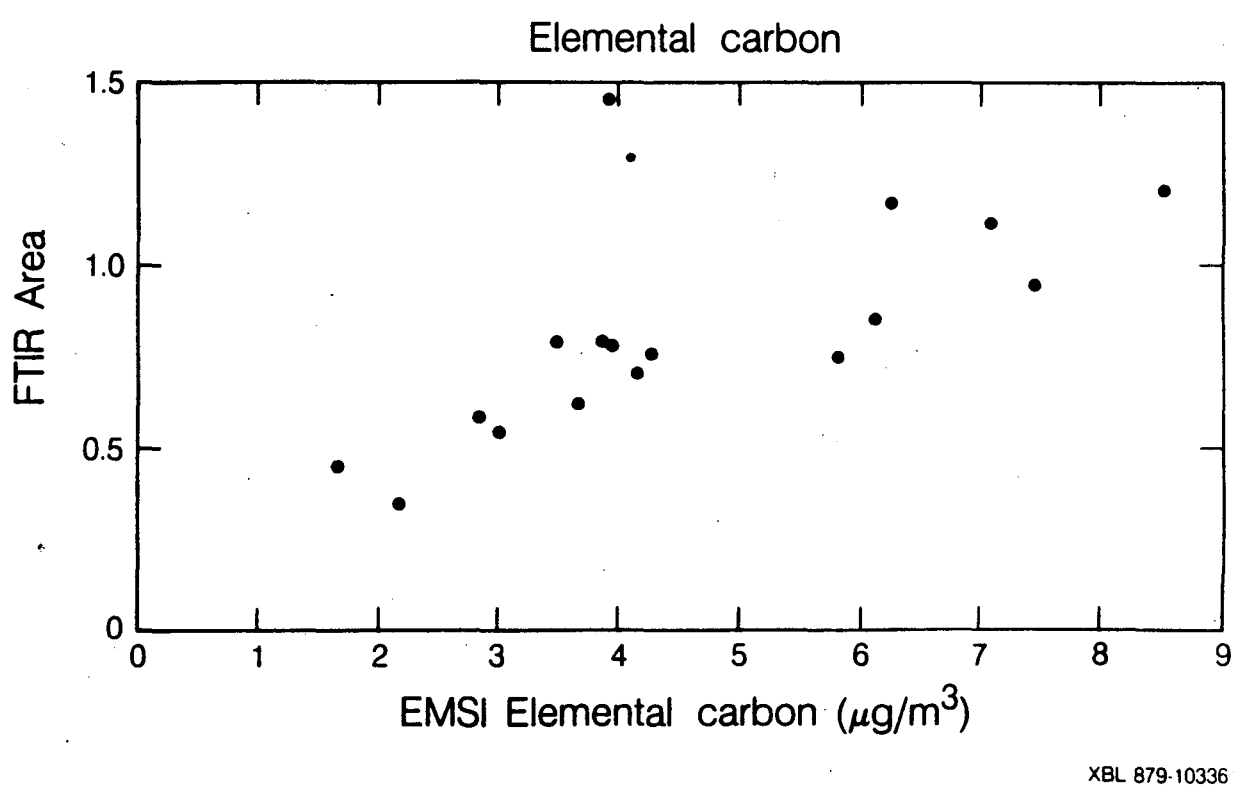

Figure 4. Scatter plot of the daily average measured area of 650 - 666 cm^{-1} region of the aerosol spectra verses the daily average ambient carbon concentration determined by thermal carbon analysis.

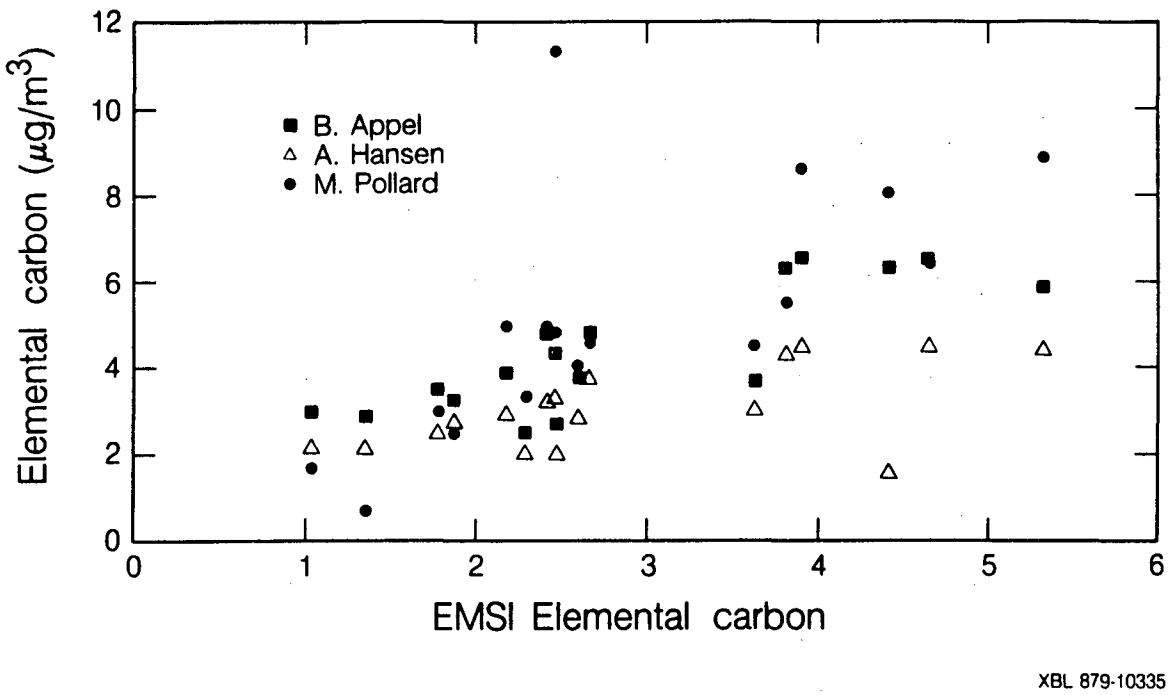

Figure 5. Average ambient carbon concentrations determined by B. Appel, A. Hansen, and M. Pollard versus the elemental carbon concentration determined by EMSI during 12-hour sampling periods.

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