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Chemical Composition of Fine Particles in the Tennessee Valley Region

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ABSTRACT

Fine particles in the atmosphere have elicited new national ambient air quality standards (NAAQS) because of their potential role in health effects and visibility-reducing haze. Since April 1997, Tennessee Valley Authority (TVA) has measured fine particles (PM_{2,5}) in the Tennessee Valley region using prototype Federal Reference Method (FRM) samplers, and results indicate that the new NAAQS annual standard will be difficult to meet in this region. The composition of many of these fine particle samples has been determined using analytical methods for elements, soluble ions, and organic and elemental carbon. The results indicate that about one-third of the measured mass is SO₄⁻², one-third is organic aerosol, and the remainder is other materials. The fraction of SO₄⁻² is highest at rural sites and during summer conditions, with greater proportions of organic aerosol in urban areas throughout the year. Additional measurements of fine particle mass and composition have been made to obtain the short-term variability of fine mass as it pertains to human exposure. Measurements to account for semi-volatile constituents of fine mass (nitrates, semi-volatile organics) indicate that the FRM may significantly under-measure organic constituents. The potentially controllable anthropogenic fraction of organic aerosols is still largely unknown.

INTRODUCTION

As the monitoring and regulatory implementation schedules for the revised PM National Ambient Air Quality Standard (NAAQS) have evolved, it has become clear that the

IMPLICATIONS

The annual standard for PM_{2.5} mass, when implemented, will likely be exceeded in much of the southeastern United States. The chemical composition must be known in order to devise effective control strategies for fine particles. This work shows that the major constituents of fine particles in the Tennessee Valley region are organics and sulfates, and thus the most effective control strategies for fine particles will likely involve controls on emissions of organics, sulfates, and their gaseous precursors.

new annual NAAQS metric for $PM_{2.5}$ will be difficult for many parts of the country to attain. The need to better understand the composition of $PM_{2.5}$ and its temporal and spatial variability is broadly supported within regulatory, industrial, and research communities. The U.S. Environmental Protection Agency (EPA) has promoted a large, three-tiered program to (in order of decreasing numbers of monitoring sites) (1) monitor fine particle mass concentrations, (2) determine the chemical composition of fine particles, and (3) evaluate new methods for determining the sources of fine particles and the details of the atmospheric processes which form them.

Proactively realizing the potential consequences of the revised PM NAAQS, the Tennessee Valley Authority (TVA) and Tennessee Valley state and local regulatory organizations began operating the first comprehensive, regional prototype Federal Reference Method (FRM) PM₂ monitoring network in the eastern United States on April 22, 1997.^{1,2} Every-third-day PM_{2.5} sampling was initiated at three core sampling stations in Nashville, Knoxville, and Lawrence County, TN. Five additional sites were added in Chattanooga and Memphis, TN (August 1997); Decatur, AL, and Paducah, KY (October 1997); and Huntsville, AL (June 1998). Analysis of the mass and chemical composition data from this network and from additional, specialpurpose studies in Chattanooga in 1998 and Nashville in 1999 is the focus of this paper. The intent of these studies was to provide an estimate of chemical composition and its spatial, seasonal, and diurnal variability, and to use these estimates to identify major components and sources of fine mass for the purpose of assisting the national effort to reduce fine particulate concentrations to which the population is exposed.

EXPERIMENTAL METHODS

The single-event fine PM sampler models used in this effort included five prototype Partisol-FRM Model 2000 (R&P Inc.) samplers, three prototype RAAS Model 2.5-100 (Graseby-Andersen) samplers, and one EPA-designated FRM PM_{2.5} sampler, Model PQ200 (BGI Inc.). Each of these samplers draws air through a 10- μ m size-selective inlet and removes particles larger than 2.5 μ m with a WINS

impactor. The $PM_{2.5}$ particles themselves were collected on Zefluor Teflon 46.2-mm filters with 2-µm pore size (Gelman Sciences, Inc.) through December 1998, and thereafter on ID-stamped Teflon filters (Whatman) with support rings. Initially, samples were collected for a 24-hr period (midnight to midnight) every third day; the sampling frequency was changed to every sixth day at most sites on October 1, 1998.

Following gravimetric analysis using a Mettler-Toledo Model MT5 microbalance, selected 24-hr samples were analyzed for elements Al through Pb using X-ray fluorescence (XRF) by EPA-approved Protocol 3. After XRF analysis, the samples were extracted ultrasonically and the TVA's Support Services Group analyzed them for NH₄⁺ by automated indophenol colorimetry and for SO₄⁻² and NO₃⁻ by ion chromatography. For selected sampling days at the network's core sites, samples of fine mass were collected on collocated samplers using quartz as the collection medium. These quartz filters were analyzed by the thermooptical reflectance (TOR) technique³ for organic and elemental carbon. The filters were then extracted ultrasonically in water and analyzed for NH_4^+ , SO_4^{-2} , and NO₃⁻ as described above for Teflon filters. Data from the collocated Teflon and quartz samples were used to determine the average chemical composition of fine particles at the three core sites in the various seasons.

During two additional periods, more intensive sampling was done at a mobile-source impacted site in Chattanooga, about 3 km from the network site. Continuous measurements of mass (tapered element oscillating microbalance [TEOM], R&P Model 1400a, with a 2.5-µm cyclone inlet, operated at 30 °C) and light scattering $(3-\lambda$ nephelometer, TSI Model 3550) were made in early March 1998, and were repeated, with the addition of continuous black carbon measurements by aethalometer⁴ (Magee Scientific, now distributed by TEII), in September 1998. The aethalometer measures the fraction of the carbonaceous aerosol that absorbs light over a broad region of the visible spectrum by determining the attenuation of light transmitted through the sample when collected on a fibrous filter. This technique has been compared with other methods for determining elemental carbon in several studies,^{5,6} most recently by Allen et al.,⁷ from which we infer an absolute accuracy of \pm ~20% for purposes of comparison with measured fine mass concentrations. The data from these measurements were used to examine diurnal and seasonal variations in mass and composition at this site, focusing on the implied influence of this variability on human exposure to fine particles.

A new sampler (PC-BOSS)⁵ designed to accurately measure both non-volatile and semi-volatile constituents of fine mass was used at urban and rural sites to test the

accuracy of the prototype FRMs. It was modified from the BYU version⁸ by addition of a mass flow meter in the major flow stream, and by addition of Visual Basic-based computer-controlled operation of valves and monitoring of flow rates. The goal was to develop and deploy an instrument that could identify the extent to which there are significant organic semi-volatile contributions to fine mass at both urban and rural locations. The organic semivolatile fraction is quantified by collecting a fine particle sample in the minor flow stream on a quartz filter downstream of a parallel-plate denuder which removed >99% of volatile organic compounds (VOCs). Any organic semivolatiles that evaporated from the filter were collected in a second stage by a carbon-impregnated paper filter (CIF). The organic carbon in particles below the cut-point of the particle concentrator (virtual impactor) is collected on a filter in the major flow stream. The fraction of semivolatiles lost is the amount of carbon found on the CIF filter divided by the sum of the carbon on minor and major flow quartz filters, corrected for the 5% losses in the concentrator. Analysis of the quartz filters was done by the DRI TOR technique,³ and analysis of the CIF filters was done by the BYU thermal evolution technique.9

RESULTS AND DISCUSSION

Our ongoing assessment of data from network operations and special studies has provided preliminary answers to the following questions.

What Are the Fine Particle Mass Concentrations in the South-Central U.S. and What Are the Implications Relative to the NAAQS?

Mass concentrations have been measured in the Tennessee Valley by FRM samplers at the sites shown in Figure 1. Fine particle annual mean mass concentrations in the Tennessee Valley range from 14 to 20 μ g/m³. Measured mass concentrations at all seven urban/suburban sites exceeded the 15 μ g/m³ level of the annual PM_{2.5} standard; only mass concentrations at the rural Lawrence County site remained below the annual standard (Table 1). Concentrations at none of the stations exceeded the 65 μ g/m³ level of the 24-hr PM_{2.5} standard. Summer-high/ winter-low seasonality in mass concentrations is evident.

How Well Does the Federal FRM Measure Fine Mass and What Positive and Negative Biases Does It Have?

The current FRM PM_{2.5} mass measurements may significantly underestimate the contribution of volatile/semivolatile nitrates and organic carbon species.⁸ Data acquired from the summer 1997 tests of the prototype PC-BOSS sampler at our Lawrence County site indicate that the undersampled, semi-volatile fraction is both highly variable and



Figure 1. Tennessee Valley PM_{2.5} monitoring network (January 2000).

significant,^{8,10} as shown in Figure 2. Further support is evident from preliminary data taken at urban sites in Nashville and Atlanta (see summary in Table 2).

VOCs including the gaseous fraction of species distributed between gas and particle phases are removed with greater than 99% efficiency by the PC-BOSS denuder. This promotes loss of collected semi-volatiles from particles collected on the quartz filter (to re-equilibrate with the gas phase), so the organic semi-volatile fraction observed with the TVA PC-BOSS sampler is an upper limit of the fraction that would be observed by a collocated FRM sampler. Even so, it is prudent to make periodic measurements that differentiate these highly variable semi-volatile and non-volatile organic carbon fractions when particle composition measurements are made, since the organic fraction of fine particles appears to be highly suspect from a health-effects standpoint. In this regard, further analysis of data from urban Nashville and Atlanta sites is in progress. We note that fine particulate NO₃⁻ can also be lost from FRM-collected filters, but since particulate NO₃⁻ levels in the southeast U.S. are low (usually <1 µg/m³ in non-urban areas), the errors caused thereby in fine mass measurements are low.

Comparison of the measured fine mass by the TEOM,¹¹ averaged over 24-hr periods, with collocated prototype-FRM measurements during the winter and summer measurement periods in Chattanooga indicate that TEOM mass was lower than FRM mass for winter measurements (Figure 3, a and b) but not for summer. This effect was observed even though the TEOM was operated at 30 °C; during the winter sampling, the average daily temperatures were in the range of 5–10 °C, but during the late summer period the average daily temperatures were 20–25 °C, only slightly lower than the TEOM filter temperature. This effect has been reported previously and is attributed to loss of semi-volatile materials from the TEOM collection filter during sampling.¹²

What Is the Composition of Fine Particles in the Tennessee Valley Region, and What Does the Composition Imply for Development of Control Strategies?

Special attention was given to determining seasonal variations in the fine particle chemical composition and composition differences between urban and rural sites. Based on composition measurements, both inorganic SO_4^{-2} and carbonaceous compounds make up large fractions of $PM_{2.5}$ mass (Figure 4). Sulfate provides the largest fraction (~50%) in background air (Lawrence County) with organic carbon

Table 1.	Monthly PM	_ mass-Tennessee	Valley	monitoring	network
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Month/Station	LC	KN	NS	CH	MP	DC	PD	HV	LR	Mean
May 97	8.9	14.8	12.9							12.2
June 97	14.3	15.8	20.8							17.0
July 97	21.8	23.7	23.7							23.1
Aug 97	15.8	19.9	22.0	22.5						20.1
Sept 97	18.1	20.6	21.7	23.2	19.1					20.6
Oct 97	13.8	15.8	19.1	24.6	16.9	18.8	16.8			18.0
Nov 97	13.2	20.1	18.0	19.9	15.6					17.4
Dec 97	13.7	19.2	20.8	21.9	17.7	23.3	18.1			19.3
Jan 98	9.8	17.5	12.4	16.6	13.3		14.1			14.0
Feb 98	12.4	9.6	17.6	15.7	22.6		16.6			15.7
March 98	11.4	11.0	16.3	14.4	15.4	15.6	13.0			13.9
April 98	14.0	13.8	16.3	16.2	16.0	22.9	15.0			16.3
May 98	16.9	26.8	22.7	24.7	26.0	22.4	23.2			23.2
June 98	13.8	14.5	16.4	16.9	18.8	23.7	15.6			17.1
July 98	15.6	22.5	22.0	22.2	18.1	24.8	24.3	24.6		21.8
Aug 98	22.7	25.4	32.8	30.3	22.0	22.4	31.8	24.8		26.3
Sept 98	18.5	23.1	21.8	26.4	20.8		19.3	19.6		21.3
Oct 98	10.5	11.8	17.2	14.9	10.5		10.6	11.4		12.8
Nov 98	15.9	16.5	16.9	26.2	15.1		23.9	20.5		19.3
Dec 98	8.3	12.9	12.5	10.6	10.7		11.2	10.6		11.0
Jan 99	10.3	12.3	13.7	13.7	9.7		13.0	11.8	6.2	11.3
Feb 99	11.3	13.4	15.1	16.3	11.5		14.7	14.3		13.8
March 99	8.8	11.9	13.1	13.1	9.5		14.6	11.3		11.8
April 99	9.7	10.9	10.8	12.9			11.7	10.3	10.0	10.9
May 99	13.9			20.2			12.5	13.5		15.0
June 99	15.0			22.2			22.2	15.3	19.9	18.9
July 99	18.5			26.3			23.5	17.6		21.5
Aug 99	21.0			30.0			22.9		24.3	24.6
Sept 99	15.7			20.2			16.7			17.5
Oct 99	14.3	15.6		19.6			16.1			16.4
Station Mean	14.3	16.8	18.2	20.1	16.3	21.7	17.5	16.1	15.1	17.3

Notes: LC = Lawrence County, TN; KN = Knoxville, TN; NS = Nashville, TN; CH = Chattanooga, TN; MP = Memphis, TN; DC = Decatur, AL; PD = Paducah, KY; HV = Huntsville, AL; LR = Look Rock, TN.

compounds making up next largest fraction (~33%). For the urban stations the situation is largely reversed, with the organic aerosol fraction being dominant (~40%), followed by SO_4^{-2} (25–35%). Control strategies designed to lower organic carbon (transportation and industrial sources) and SO_2 emissions (fossil fuel combustion sources) will therefore be more effective in achieving compliance with the PM_{2.5} annual NAAQS. Whether minor constituents contribute a disproportionate amount of the observed health effects is not addressed by this type of analysis.

What Are the Spatial, Seasonal, and Diurnal Variations in These Concentrations, and What Does This Tell Us about Sources and Fates?

Higher fractions of SO_4^{-2} were expected to be found in fine particles in the Tennessee Valley region during the summer months compared with the remainder of the year due to more stagnant synoptic air flow conditions. These conditions lead to concentration buildups for all secondary species and high rates for conversion of gaseous precursors to SO_4^{-2} and organic particulate species. Although SO_4^{-2} levels are generally higher in summer, the data shown in Figure 5 do not show the expected summer-high/ winter-low pattern in the $(NH_4)_2SO_4$ fraction of fine mass at all three core sites. This is seen only at Nashville—although the fractions are higher at the rural site in all seasons. The average molar ratio of NH_4^+ to SO_4^{-2} exceeded 1.75 in all seasons, so SO_4^{-2} fractions of fine mass were calculated as $(NH_4)_2SO_4$ for these comparisons.

Diurnal variations in several parameters relating to fine mass and its composition were observed at a mobile-source

Table 2. Summary statistics for TVA's PC-BOSS-Atlanta Supersite, August 1999.

Property	Average	Standard Deviation	N of Values
Mass	26.5	13.6	42
Weighted Mass	24.7	10.9	(57)
SO4 ⁻²	8.70	4.05	38
NVOC	5.1	2.5	37
EC	1.76	0.87	37
Minor SO₄ ⁻² /Total	0.69	0.08	38
Minor NH₄+/Total	0.69	0.12	38
Molar NH4 ⁺ /SO ⁻²	1.76	0.16	40
Volatile OC/TotaI	0.14	0.09	34
(NH ₄) ₂ SO ₄ Fr of Mass	0.49	0.12	38
NVOC Fr of Mass	0.31	0.16	36

Note: All concentrations in units of µg/m³.

the network site during intensive sampling periods in late February-early March 1998 and in September 1998. Hourly average values for TEOM PM_{2.5} mass, 550-nm (green) light scattering by nephelometry and, in September 1998, continuous elemental (black) carbon measurements using an aethalometer⁴ were calculated (see Figures 6 and 7). As noted above, TEOM mass for the late winter measurements were estimated to under-measure the mass by about 40% due to particle evaporation from the TEOM filters (Figure 3a). The aethalometer measures black carbon, and comparisons with other elemental carbon measurements of integrated samples by thermal techniques indicate that the accuracy of the aethalometer estimate of elemental carbon is on the order of $\pm 30\%$. The hourly average data can be used to evaluate diurnal trends, however, even with the accuracy limitations. The observed diurnal trends shown in Figures 6 and 7 show the effects of primary sources and meteorology. Error bars are the between-day variability in the averages for each hour. Higher concentrations of primary particles (e.g.,

elemental carbon, Figure 6) and, to a lesser extent, mass were observed during the morning rush hour when the shallow nighttime boundary layer had not yet broken up, but not during the late-afternoon high-traffic period. Higher concentrations of mass and black carbon and higher light scattering coefficients were observed at night, when a stable

impacted site in Chattanooga, located about 3 km from

nocturnal boundary layer was usually present, than during well-mixed conditions in late morning and afternoon (1100–1500 hr, Figures 6 and 7), presumably due to poor vertical mixing with layers above in the vertical.

What Are the Controllable Fractions of Fine Mass and What Are the Sources of Those Potentially Controllable Fractions?

The largest fractions of fine mass observed in the Tennessee Valley are attributable to organic carbonaceous material and $(NH_4)_2SO_4$. The remainder of the fine mass (elemental carbon, soil-derived components, and trace metals/elements) comprises minor portions. The SO_4^{-2} fraction can, in theory, be controlled by further reducing emissions of its gaseous precursor, SO_2 , although nonlinear gas-to-particle conversion processes appear to be significantly reducing the "bang for the buck." The organic fraction is largely uncharacterized, and a high priority should be placed on characterizing what fraction of it is controllable by reducing man-made emissions of particulate organics and their gas-phase precursors.

A major portion of the fine mass in the Tennessee Valley is regionally transported, leading to a high correlation of mass levels between network sites. An example of extraregionally transported fine mass is available from data taken during the "Mexican smoke" episode of May 1998. Fine mass was elevated throughout the network for multiple



Figure 2. Semi-volatile organics as a fraction of organic carbon by PC-BOSS.



Figure 3a. Comparison of TEOM and FRM 24-hr mass in Chattanooga, TN, February-March 1998.



Figure 3b. Comparison of TEOM and FRM 24-hr mass in Chattanooga, TN, September 1998.



Figure 4. Average PM_{2.5} composition for network core sites.

sampling days, and it was enriched in fine potassium (and, to a lesser extent, fine Si and Ca) to a much greater extent than in SO_4^{-2} and other ions during this period. This suggests that fine particles have long lifetimes, and that region-wide controls of precursors to organics and sulfates may be needed to reduce average fine mass concentrations. Indeed, year-to-year differences in meteorological conditions may be crucial in determining fine particle concen-

trations and in establishing which strategies are likely to be successful in reducing those concentrations.

CONCLUSIONS

Since April 1997, TVA has measured PM_{2.5} in the Tennessee Valley region using prototype FRM samplers, and results indicate that compliance with the new NAAQS annual standard will be difficult. The chemical composition of fine

particle samples has been estimated, and our results indicate that about 30-50% of the measured mass is (NH₄)₂SO₄, about onethird is organic PM, and the remainder is other materials. The SO_4^{-2} fraction is highest at rural sites and during stagnant summer meteorological conditions, with large fractions of organic aerosol mass in urban areas. Short-term variability of fine particle mass has been measured, and sampling that has been performed accounts for semivolatile constituents of fine mass (nitrates, organics). Results show diurnal variability affecting exposure and suggest that FRM measurements may significantly underestimate organic constituents. Potentially controllable anthropogenic sources of fine particulate organics remain largely uncharacterized.



Figure 5. Calculated $(NH_{d})_2SO_4$ as fraction of fine mass, by season.



Figure 6. Diurnal variations in TEOM mass and light scattering, Chattanooga, TN, February–March 1998.



Figure 7. Diurnal variations in TEOM mass, black carbon and light scattering, Chattanooga, TN, September 1998.

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