Draft 3

Guideline on Speciated Particulate Monitoring

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1.0 INTRODUCTION

Chemical speciation of suspended particulate matter (PM) is needed for the U.S. Environmental Protection Agency's (EPA) $PM_{2.5}$ (mass fraction of particles with aerodynamic diameters less than 2.5 μm) monitoring program as set forth in 40 CFR part 58, "Ambient Air Quality Surveillance for Particulate Matter" (U.S. Environmental Protection Agency, 1997a). Within one year from the NAAQS effective date (September 16, 1997), chemical speciation will be initiated at 50 $PM_{2.5}$ core (community representative) sites. Approximately 25 of these $PM_{2.5}$ core sites are to be collocated with Photochemical Air Monitoring Stations (PAMS) that measure ozone and its precursors. $PM_{2.5}$ filter samples that require chemical speciation will be collected with aerosol samplers and on sampling schedules approved by the U.S. EPA. At a minimum, chemical speciation will quantify significant $PM_{2.5}$ components of geological material, sulfate, nitrate, ammonium, organic carbon, and elemental carbon, in addition to mass concentrations. Many of these sites will also measure PM_{10} (mass fraction of particles with aerodynamic diameters less than 10 μm) for which chemical speciation may be desirable, but not required.

The 50 speciation sites constitute the initial chemical speciation monitoring network and will be selected by the U.S. EPA in consultation with regional and state administrators. The chemical speciation network is part of the National Ambient Monitoring Stations (NAMS) network and will provide national consistency for trend analysis and will serve as a model for other speciation efforts (U.S. EPA, 1997a). U.S. EPA plans to expand this initial chemical speciation network of 50 sites to include 300 sites and tailor the sampling and analysis of chemically speciated data to the needs of specific geographical areas. Other government, academic, and commercial entities may further increase the number of long-term speciated monitoring sites.

1.1 Background

On July 18, 1997, the U.S. Environmental Protection Agency promulgated the new National Ambient Air Quality Standards (NAAQS) for particulate matter (PM) (U.S. Environmental Protection Agency, 1997b). The NAAQS applies to the mass concentration of particles with aerodynamic diameters lower than 2.5 μm ($PM_{2.5}$) and 10 μm (PM_{10}). The NAAQS specify:

- Twenty-four hour average $PM_{2.5}$ not to exceed 65 $\mu g/m^3$ for a three-year average of annual 98th percentiles at any population-oriented monitoring site in a Metropolitan Planning Area (MPA).

- Three-year annual average $PM_{2.5}$ not to exceed 15 $\mu g/m^3$ concentrations from a single community-oriented monitoring site or the spatial average of eligible community exposure sites in a MPA.

- Twenty-four hour average PM_{10} not to exceed 150 $\mu g/m^3$ for a three-year average of annual 99th percentiles at any monitoring site in a monitoring area.

- Three-year average PM_{10} not to exceed 50 $\mu g/m^3$ for three annual average concentrations at any monitoring site in a monitoring area.

When the PM_{10} NAAQS (U.S. EPA, 1987) were reevaluated (U.S. EPA, 1996), it was recognized that $PM_{2.5}$ and PM_{10} mass concentrations are indicators of adverse health effects, not necessarily the exact causes of those effects. Chemical speciation was deemed essential for establishing more specific relationships between particle concentrations and measures of public health. Chemical speciation also facilitates understanding of PM temporal and spatial variations, source/receptor relationships, and the effectiveness of emissions reduction strategies. Establishment of a chemical speciation monitoring network and interpretation of the resulting mass and speciated chemical data will prepare the scientific community for the next review of the NAAQS and revision of the air quality criteria document.

1.2 Objectives

To support these measurements, this guidance intends to:

- Describe $PM_{2.5}$ sampling equipment and methods that acquire filter deposits amenable to different chemical analyses, including Federal Reference Methods (FRM), Federal Equivalent Methods (FEM), and IMPROVE (Interagency Monitoring of Protected Visual Environments) methods.

- Identify artifacts and interferences that result from filter sampling and handling and describe methods to minimize them.

- Associate chemical components found in suspended particles with analytical methods to quantify them and describe how those methods can be efficiently and accurately applied to many samples.

- Evaluate the feasibility and practicality of different sampling and analysis combinations for a variety of monitoring situations and data uses.

- Specify procedures for unifying field sampling and laboratory measurements to obtain data sets of defined accuracy, precision, validity, and equivalence.

1.3 Related Documents

This chemical speciation guidance document builds upon past and current U.S. EPA documents, including:

- U.S. Environmental Protection Agency (1994). Guidelines for PM_{10} Sampling and Analysis Applicable to Receptor Modeling. EPA-452/R-94-009. U.S. EPA Office of Air Quality Planning and Standards, Research Triangle Park, NC. March 1994.

- J.G. Watson et al. (1997a). Guidance for Network Design and Optimum Site Exposure for $PM_{2.5}$ and PM_{10} – Draft Version 3. Prepared for U.S. EPA Office of Air Quality Planning and Standards, Research Triangle Park, NC, by Desert Research

Institute, Reno, NV. September 19, 1997.

- M. Pitchford et al. (1997). Prototype $PM_{2.5}$ Federal Reference Method Field Studies Report – An EPA Staff Report. U.S. EPA Office of Air Quality Planning and Standards, Las Vegas, NV. July 9, 1997.

- J.G. Watson et al. (1997b). Guidance for Using Continuous Monitors in $PM_{2.5}$ Monitoring Networks. In preparation for U.S. EPA Office of Air Quality Planning and Standards, Research Triangle Park, NC, by Desert Research Institute, Reno, NV.

- U.S. EPA (1997a). Revised Requirements for Designation of Reference and Equivalent Methods for $PM_{2.5}$ and Ambient Air Quality Surveillance for Particulate Matter – Final Rule. 40 CFR part 58. *Federal Register*, **62**(138):38830-38854. July 18, 1997.

- U.S. EPA (1997b). Revised Requirements for Designation of Reference and Equivalent Methods for $PM_{2.5}$ and Ambient Air Quality Surveillance for Particulate Matter – Final Rule. 40 CFR part 53. *Federal Register*, **62**(138):38763-38830. July 18, 1997.

- U.S. EPA (1997c). National Ambient Air Quality Standards for Particulate Matter – Final Rule. 40 CFR part 50. *Federal Register*, **62**(138):38651-38760. July 18, 1997.

- U.S. EPA (1997d). National Ambient Air Quality Standards for Particulate Matter; Availability of Supplemental Information and Request for Comments – Final Rule. 40 CFR part 50. *Federal Register*, **62**(138):38761-38762. July 18, 1997.

1.4 Guide to Document

This section states the background and objectives of this PM sampling and analysis guidance document. Section 2 describes the chemical and physical properties of PM and its major chemical components. Existing filter-based PM sampling and analysis methods are summarized in Sections 3 and 4, respectively. Major concerns with respect to PM sampling artifacts and interferences are identified in Section 5. Components of quality assurance and quality control are specified in Section 6. The validity of the environmental measurements is defined in Section 7. Section 8 outlines the approaches to field- and laboratory-integrated monitoring strategies in order to address source/receptor relationships. A document summary is provided in Section 9. Cited references and resources that provide more detail on specific topics are assembled in Section 10.

2.0 PHYSICS AND CHEMISTRY OF ATMOSPHERIC PARTICLES

Particles in the atmosphere can be characterized by their size, composition, shape, color, number, and gas/particle phase equilibrium. It is not useful, nor does technology exist, to measure every aspect of these properties. Previous studies of chemically characterized suspended particles in source and receptor samples from a variety of environments (e.g., Lioy et al., 1980; Chow and Watson, 1989; Watson and Chow, 1992) identify several aerosol properties related to excessive concentrations, pollution sources, visibility, and health.

2.1 Particle Size Distributions

Major features of particle size mass distribution found in the atmosphere are illustrated in Figure 2-1. The “nucleation” range, also termed “ultrafine particles” (Oberdörster et al., 1995; Fitzgerald et al., 1997; Kotzick et al., 1997) consists of particles with diameters less than ~0.08 µm that are emitted directly from combustion sources or that condense from cooled gases soon after emission. Nucleation particle lifetimes are usually less than one hour because they rapidly coagulate with larger particles or serve as nuclei for cloud or fog droplets. The nucleation range is detected only when fresh emissions are close to a measurement site or when new particles have been recently formed in the atmosphere (Lundgren and Burton, 1995).

The “accumulation” range consists of particles with diameters between 0.08 and ~2 µm. These particles result from the coagulation of smaller particles emitted from combustion sources, from gas-to-particle conversion, from condensation of volatile species, and from finely ground dust particles. Nucleation and accumulation ranges constitute the “fine” particle size fraction, and the majority of sulfuric acid, ammonium bisulfate, ammonium sulfate, ammonium nitrate, organic carbon, and elemental carbon is found in this size range. Particles larger than ~2 or 3 µm are called “coarse particles” that result from grinding activities and are dominated by material of geological origin. Pollen and spores also inhabit the coarse particle size range, as do ground up trash, leaves, and tires. Particles at the low end of the coarse size range also occur when cloud and fog droplets form in a polluted environment, then dry out after having scavenged other particles and gases (Jacob et al., 1986).

The $PM_{2.5}$, PM_{10}, and TSP (Total Suspended Particulate) size fractions commonly measured by air quality monitors are identified in Figure 2-1 by the portion of the size spectrum that they occupy. The mass collected is proportional to the area under the distribution within each size range. The TSP size fraction ranges from 0 to ~40 µm, the PM_{10} fraction ranges from 0 to 10 µm, and the $PM_{2.5}$ size fraction ranges from 0 to 2.5 µm in aerodynamic diameter. No sampling device operates as a step function, passing 100% of all particles below a certain size and excluding 100% of the particles larger than that size. When sampled, each of these size ranges contains a certain abundance of particles above the upper size designation of each range (Watson et al., 1983; Wedding and Carney, 1983).

Figure 2-1 shows the accumulation range to consist of at least two sub-modes (Watson and Chow, 1994a), which differs from many other published literature that show only a single peak in this region (Whitby et al., 1972). Recent measurements of chemical-specific size distributions show that these sub-modes exist in several different environments (Hering and Friedlander, 1982; Hoppel et al., 1990; Sloane et al., 1991). John et al. (1990) interpreted the peak centered at ~0.2 µm as a "condensation" mode containing gas-phase reaction products, and the ~0.7 µm peak as a "droplet" mode resulting from growth by nucleation of particles in the smaller size ranges and by reactions that take place in water droplets. The liquid water content of ammonium nitrate, ammonium sulfate, sodium chloride, and other soluble species increases with relative humidity, and this is especially important when relative humidity exceeds 70% (Rogers and Watson, 1991). When these modes contain soluble particles, their peaks shift toward larger diameters as humidity increases (Tang, 1976, 1980, 1993; Tang et al., 1977; McMurry et al., 1987; Zhang, 1989).

The peak of the coarse mode may shift between ~6 and 25 µm (Lundgren and Burton, 1995). A small shift in the 50% cut-point of a PM_{10} sampler has a large influence on the mass collected because the coarse mode usually peaks near 10 µm. On the other hand, a similar shift in cut-point near 2.5 µm results in a small effect on the mass collected owing to the low quantities of particles in the 1 to 3 µm size range (Chow, 1995; Watson, 1995).

Figure 2-2 shows the size distribution of suspended particles measured from common emission sources. Construction dusts, road dusts, and soil dusts formed from grinding down of larger particles are predominantly in the coarse particle size range, with minor or moderate quantities in the $PM_{2.5}$ fraction. Combustion particles, on the other hand, dominate the $PM_{2.5}$ size fraction. Chemical components that distinguish between geological dusts, combustion products, and secondary aerosols can be used to effectively classify TSP (total suspended particles) or PM_{10} mass concentrations into accumulation or coarse fractions of the particle size distribution.

2.2 Major Chemical Components

Most of the $PM_{2.5}$ or PM_{10} mass in urban and nonurban areas can be explained by a combination of the following chemical components:

- **Geological Material:** Suspended dust consists mainly of oxides of aluminum, silicon, calcium, titanium, iron, and other metal oxides (Chow and Watson, 1992. The precise combination of these minerals depends on the geology of the area and industrial processes such as steel-making, smelting, mining, and cement production. Geological material is mostly in the coarse particle fraction (Houck et al., 1990), and typically constitutes ~50% of PM_{10} while only contributing 5 to 15% of $PM_{2.5}$ (Watson et al., 1995a)

- **Sulfate:** Ammonium sulfate ($(NH_4)_2SO_4$), ammonium bisulfate ($(NH_4HSO_4$), and sulfuric acid (H_2SO_4) are the most common forms of sulfate found in atmospheric particles, resulting from conversion of gases to particles as described below. These compounds are water-soluble and reside almost exclusively in the $PM_{2.5}$ size fraction. Sodium sulfate (Na_2SO_4) may be found in coastal areas where sulfuric acid has been neutralized by sodium chloride (NaCl) in sea salt. Though gypsum (Ca_2SO_4) and some other geological compounds contain sulfate, these are not easily dissolved in water for chemical analysis, are more abundant in the coarse fraction than in $PM_{2.5}$, and they are usually classified in the geological fraction.

- **Nitrate:** Ammonium nitrate (NH_4NO_3) is the most abundant nitrate compound, resulting from a reversible gas/particle equilibrium between ammonia gas (NH_3), nitric acid gas (HNO_3), and particulate ammonium nitrate. Because this equilibrium is reversible, ammonium nitrate particles can easily evaporate in the atmosphere, or after they have been collected on a filter, owing to changes in temperature and relative humidity (Stelson et al., 1982; Watson et al., 1994a). Sodium nitrate ($NaNO_3$) is found in the $PM_{2.5}$ and coarse fractions near sea coasts and salt playas (e.g., Watson et al., 1995a) where nitric acid vapor irreversibly reacts with sea salt (NaCl).

- **Ammonium:** Ammonium sulfate ($(NH_4)_2SO_4$), ammonium bisulfate (NH_4HSO_4), and ammonium nitrate (NH_4NO_3) are the most common compounds containing ammonium from irreversible reactions between sulfuric acid and ammonia gas. Figure 2 -3 from Chow et al. (1996a) shows an example in which sulfate is primarily present as neutralized ammonium sulfate, while ammonium bisulfate and sulfuric acid are not present in great abundances as evidenced by slopes that approach unity when ammonium sulfate is assumed. The effect of marine aerosol is also shown in this example.

- **Sodium Chloride:** Salt is found in suspended particles near sea coasts, open playas, and after de-icing materials are applied. In its raw form (e.g., de-icing sand), salt is usually in the coarse particle fraction and classified as a geological material. After evaporating from a suspended water droplet (as in sea salt or when resuspended from melting snow), it is abundant in the $PM_{2.5}$ fraction. As noted above, sodium chloride is often neutralized by nitric or sulfuric acid and is classified as a sulfate or nitrate.

- **Organic Carbon:** Particulate organic carbon consists of hundreds, possibly thousands, of separate compounds that contain more than 20 carbon atoms ($>C_{20}$). Table 2-1 identifies several of the compounds that have been measured in suspended particles, but these constitute no more than 10% measured organic carbon (Rogge et al., 1991). Because of this lack of molecular specificity, and owing to the semi-volatile nature of many carbon compounds (C_{20} to C_{40}), particulate "organic carbon" is operationally defined by the sampling and analysis method. Differences caused by this operational definition are discussed later in this document.

- **Elemental Carbon:** Elemental carbon is black, often called "soot." Elemental carbon contains pure, graphitic carbon, but it also contains high molecular weight, dark-colored, non-volatile organic materials such as tar, biogenics, and coke. Particulate "elemental carbon" is also operationally defined, as discussed later in this document.

- **Liquid Water:** Soluble nitrates, sulfates, ammonium, sodium, other inorganic ions, and some organic material (Saxena and Hildemann, 1996) absorb water vapor from the atmosphere, especially when relative humidity exceeds 70%. Sulfuric acid absorbs some water at all humidities. Particles containing these compounds grow into the droplet mode as they take on liquid water. Some of this water is retained when particles are sampled and weighed for mass concentration. The precise amount of water quantified in a $PM_{2.5}$ depends on its ionic composition and the equilibration relative humidity applied prior to laboratory weighing.

Tables 2-2 and 2-3 summarize concentrations of the most abundant and most commonly measured chemical components, typical of those to be acquired by U.S. EPA's chemical speciation network, for PM_{10} and $PM_{2.5}$ from several locations and times of year. Chemical-specific PM_{10} measurements are more abundant than $PM_{2.5}$ mass concentrations (e.g., Chow et al., 1992a, 1993a; Watson et al., 1997c). However, since the majority of the sulfates, nitrates, ammonium, and carbon are in the $PM_{2.5}$ fraction, the non-geological PM_{10} concentrations in Table 2-2 provide substantial information about the nature of $PM_{2.5}$ at those sites.

Organic carbon and/or nitrate are the most abundant species in PM_{10} and $PM_{2.5}$ at all locations reported in Tables 2-2 and 2-3. In coastal environments, sodium and nitrate are highly enriched in the PM_{10} fraction as compared to the $PM_{2.5}$ fraction, implying the presence of sodium nitrate in coarse particles. This phenomenon presumably occurs because nitric acid reacts with sea salt (Pilinis and Seinfeld, 1987). PM_{10} and $PM_{2.5}$ particle chloride is also enriched at the coastal sites, with average PM_{10} chloride concentrations exceeding 1 or 2 $\mu g/m^3$ (Chow et al., 1994a). Chow et al. (1996a) show that sodium chloride constitutes over 50% of PM_{10} and 30% of $PM_{2.5}$ at Point Reyes on the California coast (five to ten times higher than usually found at inland sites).

Sodium, aluminum, silicon, potassium, calcium, iron, and zinc are abundant only in the coarse particle fraction (PM_{10} minus $PM_{2.5}$), consistent with expected contributions from marine aerosol (e.g., sodium) and suspended dust (e.g., aluminum, silicon, calcium, iron). The proportion of geological material in PM_{10} varies from site to site, with over 80% of PM_{10} attributable to geological material in Las Vegas, NV (Chow et al., 1995a; Chow and Watson, 1997a) and less than 20% in San Jose, CA (Chow et al., 1995b).

In Table 2-3, average summer 1990 $PM_{2.5}$ concentrations at regionally representative sites in central California show simultaneous mass concentrations ranging from <3 $\mu g/m^3$ at Point Reyes, CA (a coastal transport site) to almost 50 $\mu g/m^3$ at the Edison site (located downwind of urban sources and agricultural operations at the southern end of California's San Joaquin Valley). Organic carbon, elemental carbon, nitrate, sulfate, and ammonium are almost always the major components in $PM_{2.5}$ (Chow et al., 1994a; Chow et al., 1996a). Geological material abundances in $PM_{2.5}$ typically range from 5% to 15%, but they could be as high as 30% to 40% of $PM_{2.5}$ at Central California sites (e.g., Crow's Landing, Edison) during summer (Chow et al., 1996a).

In the western parts of the U.S., $PM_{2.5}$ or PM_{10} appear to be higher during fall or winter than during the summer. Sulfate, however, shows higher summer averages than those during the fall at six Southern California monitoring sites (Chow et al., 1994a). $PM_{2.5}$ organic carbon is most often enriched during the fall and winter in residential neighborhoods. PM_{10} organic carbon was highest at the U.S./Mexican border where motor vehicle exhaust, field burning, and cooking were the major emission sources.

2.3 Properties that Quantify Source Contributions

The relative abundance of chemical components in an ambient $PM_{2.5}$ sample reflects the chemical composition of the source emissions in the monitored environment. Chemical source profiles are the fractional mass abundances of measured chemical species relative to primary $PM_{2.5}$ mass in source emissions.

Figures 2-4 through 2-7 show source profile examples for the common $PM_{2.5}$ emitters of: (1) geological material, (2) motor vehicle exhaust, (3) wood and coal burning, and (4) coal-fired power generators (Watson et al., 1996a). In each of these illustrations the height of each bar indicates the average fractional abundance for the indicated chemical, while the dot shows the standard deviation of the average. When the height of the bar exceeds the position of the dot, and when the height of the bar is much higher than it is in other profiles, the corresponding species is considered as a good marker for that source type.

Figures 2-4 through 2-7 also include the ratio of gaseous sulfur dioxide (SO_2) and gaseous ammonia (NH_3) to $PM_{2.5}$ mass emissions. Sulfur dioxide and ammonia are important precursors to secondary aerosol (e.g., ammonium sulfate, ammonium bisulfate, ammonium nitrate), and their chemical abundances are useful at source and receptor locations to determine the causes of high $PM_{2.5}$ levels.

Previous chemical profile compilations (e.g., Watson, 1979; Shareef et al., 1984; Sheffield and Gordon, 1985; Core and Houck, 1987; Cooper et al., 1987; Houck et al., 1989a, 1989b, 1989c, 1989d, 1989e; Chow and Watson, 1994b; Watson and Chow, 1994b; Watson et al., 1994a, 1996a-b; Chow and Watson, 1997b-c; Chow et al., 1997a) include chemical abundances of elements, ions, and carbon for geological material (e.g., paved and unpaved road dust, soil dust, storage pile), motor vehicle exhaust (e.g., diesel-, leaded-gasoline-, and unleaded-gasoline-fueled vehicles), vegetative burning (e.g., wood stoves, fireplaces, forest fires, and prescribed burning), industrial boiler emissions, and other aerosol sources. More modern, research-oriented profiles include specific organic compounds or functional groups, elemental isotopes, and microscopic characteristics of single particles.

As fuels, technologies, and use patterns have changed from 1970 to the present, so have the chemical profiles for many emissions sources. Lead has been phased out of U.S. and Canadian fuels, but it is still used in some Mexican gasolines that might affect $PM_{2.5}$ in border areas. Catalytic converters on spark-ignition vehicles, improved compression-ignition engines (Pierson et al., 1996), and newly-designed wood combustion appliances (Myren, 1992) have substantially reduced carbon abundances in emissions from these small but numerous sources. Similarly, process improvements and new source performance standards have resulted in changes in chemical component emissions from large industrial emitters. Source profiles must be paired in time with ambient $PM_{2.5}$ chemical species measurements to establish a reasonable estimate of what is expected in ambient air. Figures 2-4 through 2-7 from northwestern Colorado (Watson et al., 1996a) represent the most recent (i.e., 1995), but by no means the most complete, $PM_{2.5}$ emissions compositions. These differ substantially from chemical source profiles measured as recently as 1990.

Figure 2-4 shows the similarities and differences among chemical abundances in three sub-types of $PM_{2.5}$ geological emitters: (1) paved road dust, (2) unpaved road dust, and (3) natural soils. Aluminum (Al), silicon (Si), potassium (K), calcium (Ca), and iron (Fe) have large abundances with low variabilities. The total potassium (K) abundance is 15 to 30 times the abundance of soluble potassium (K^+). Aluminum (Al), potassium (K), calcium (Ca), and iron (Fe) abundances are similar among the profiles, but the silicon (Si) abundances range from 14% in unpaved road dust to 20% in paved road dust.

Lead (Pb) is most abundant in paved road dust, and is as low as 0.004% in the other geological profiles, probably due to deposition from previously emitted leaded-gasoline vehicle exhaust or remnants of lead from the exhaust trains of older vehicles. Elemental carbon (EC) abundances are highly variable in geological material, and are often negligible in natural soil samples. Organic carbon (OC) is typically 5% to 15% in geological emitters. It is most abundant in paved road and agricultural dusts, although the specific compounds are probably quite different for these two sources (Chow et al., 1994b).

Motor vehicle emissions (e.g., brake and tire wear, oil drips) could result in greater abundances of Pb, EC, and OC in paved road dust. Soluble sulfate, nitrate, and ammonium abundances are low, in the range of 0 to 0.3%. Sodium and chloride are also low, with less than 0.5% in abundance. Larger abundances of these materials may be found temporarily soon after roadway de-icing, however.

Mobile source $PM_{2.5}$ emissions determined from roadside sampling are shown in Figure 2-5 for: (1) local traffic emissions, (2) highway vehicle emissions, and (3) composite vehicle fleet emissions. Organic and elemental carbon are the most abundant species in motor vehicle exhaust, accounting for over 95% of the total mass. The lead (Pb) abundance is negligible and highly variable (0.024 ± 0.036%) in motor vehicle exhaust profiles. The abundance of bromine (Br) is also low, in the range of 0.01% to 0.05%. Zinc is present in most exhaust profiles, usually at levels of 0.05% or less. The abundance of chloride is 1.5% to 3.5%.

As shown in Figure 2-5, organic carbon abundances ranges from 36% in highway vehicle emissions to 70% in local traffic emissions. The ratio of organic to total carbon (OC/TC) is 0.58 in the composite vehicle profile for northwestern Colorado. This OC/TC ratio is similar to those reported by Watson et al. (1994b) in Phoenix, AZ, with 0.69 for gasoline-fueled vehicle exhaust, 0.55 for diesel-fueled vehicle exhaust, and 0.52 for a mixture of vehicle types in roadside tests. Earlier measurements in Denver, CO (Watson et al., 1990) reported an OC/TC ratio of 0.39 for the cold transient cycle and 0.81 for the cold stabilized cycle.

Figure 2-6 compares residential wood combustion (RWC) residential coal combustion (RCC), and forest fire $PM_{2.5}$ profiles. Average OC abundances range from ~50% in RWC and the forest fire profiles to ~70% in the RCC profile. EC averages 3% in forest fire, 12% in RWC, and 26% in RCC. Note that the OC/TC ratio is highest in the forest fire profile (OC/TC = 0.94) and similar for the two residential combustion profiles, with 0.73 in RCC and 0.81 in RWC. Chow and Watson (1997c) measured profiles for asparagus field burning in California's Imperial Valley with OC/TC ratios of 0.93, similar to the 0.94 ratio found in the forest fire emissions. A similar observation was made for charbroil cooking emissions, with 60% to 70% OC abundances and high (>0.95) OC/TC ratios.

The K^+/K ratios of 0.80 to 0.90 in these burning profiles (Calloway et al., 1989) are in large contrast to the low soluble to total potassium ratios found in geological material. Sulfate, nitrate, and silicon abundances in RCC are 2 to 4 times those in the RWC and forest fire profiles. The ammonium abundance is highly variable, with an average of 1.4% in RCC and 0.1% in the RWC and forest fire profiles. Sulfur dioxide gas, a particulate sulfate precursor, can also be ratioed to $PM_{2.5}$ emissions. Figure 2-6 shows that the SO_2 abundance is negligible in the RWC and forest fire profiles. Only 50% of the RCC samples reported significant $SO_2/PM_{2.5}$ mass ratios. These ratios are highly variable, ranging from 112 ± 3% to 532 ± 27%. Sulfate in RCC profiles is ~3%. Selenium (Se) is also detected in RCC emissions.

Coal-fired power generation profiles shown in Figure 2-7 differ substantially, even though the fuels are similar, owing to the different emissions control technologies. The example shown in Figure 2-7 includes: (1) Unit 1 equipped with an electrostatic precipitator (ESP) and ammonia injection; (2) Unit 2 equipped with ESP and a wet scrubber; and (3) Unit 3 equipped with a fabric baghouse and dry lime scrubber. Sulfate is one of the most abundant constituents in the particle phase (3% in Unit 1, 13% in Unit 3, and 23% in Unit 2). EC in Unit 2 (8%) is higher than in Unit 1 (4%) and Unit 3 (1%). The abundances of OC are highly variable (~2% in Units 2 and 3 and 34% in Unit 1). OC/TC ratios averaged 0.22 in Unit 3, 0.69 in Unit 3, and 0.89 in Unit 1.

Crustal elements such as silicon (Si), calcium (Ca), and iron (Fe) in the coal-fired boiler profiles are present at 30% to 50% of the corresponding levels in geological material with the exception of aluminum (Al) which is present at similar or higher levels than those found in geological material. Other elements such as phosphorus (P), potassium (K), titanium (Ti), chromium (Cr), manganese (Mn), strontium (Sr), zirconium (Zr), and barium (Ba) are present at less than 1% levels.

Selenium (Se) is detected at the level of 0.2% to 0.4% in Units 1 and 2, but not found in Unit 3. Selenium is usually in the gaseous phase within hot stack emissions, and it condenses on particles when air is cooled in the dilution chamber. Abundances of calcium (15%), chloride (1%), and nitrate (1%) in Unit 3 are a few times higher than in Units 1 and 2. These differences may have resulted from the dry lime scrubber present in Unit 3. Sulfur dioxide in these coal-fired boiler emissions are highly variable and orders of magnitude higher than those found in the residential coal combustion profile. Ammonia is detectable (7% to 10%) in coal-fired boiler emissions from Units 2 and 3, and is 3,365% in Unit 1 due to ammonia injection.

The variability of $PM_{2.5}$ profiles illustrated in Figures 2-4 through 2-7 provide the following insights:

- Source emissions of precursor gaseous and primary particles are highly variable due to differences in fuel use, operating conditions, and sampling methods. Source and ambient measurements must be paired in time to establish reasonable estimates of source/receptor relationships.

- Measurements of seven major components discussed in Section 2.2 for the $PM_{2.5}$

speciated monitoring network can only provide a first-order source attribution of ambient $PM_{2.5}$ concentrations to major source types. Additional measurements of precursor gases, isotopes, particle morphology, and organics need to be acquired to quantitatively assess the associated source sub-types.

- Trace metals acquired from elemental analysis of Teflon-membrane filters are only abundant in the geological profiles. Identifying and quantifying major source contributions are not sufficient for source attribution; chemical speciation of ammonium, sulfate, nitrate, organic carbon, and elemental carbon are also essential.

Sulfate, nitrate, and ammonium abundances in these directly emitted particles are not sufficient to account for the concentrations of these species measured in the atmosphere. Ambient mass concentrations contain both primary and secondary particles. Primary particles are those which are directly emitted by sources; these particles often undergo few changes between source and receptor. Atmospheric concentrations of primary particles are, on average, proportional to the quantities that are emitted. Secondary particles are those that form in the atmosphere from gases that are directly emitted by sources.

Sulfur dioxide, ammonia, and oxides of nitrogen are the precursors for sulfuric acid, ammonium bisulfate, ammonium sulfate, and ammonium nitrate particles (Seinfeld, 1986; Watson et al., 1994a). Several volatile organic compounds (VOC) may also change into particles; the majority of these transformations result from intense photochemical reactions that also create high ozone levels (Grosjean and Seinfeld, 1989). Secondary particles usually form over several hours or days and attain aerodynamic diameters between 0.1 and 1 µm, as shown in Figure 2-1. Several of these particles, notably those containing ammonium nitrate, are volatile and transfer mass between the gas and particle phase to maintain a chemical equilibrium (Stelson and Seinfeld, 1982a-c). This volatility has implications for ambient concentration measurements as well as for gas and particle concentrations in the atmosphere.

Ambient concentrations of secondary aerosols are not necessarily proportional to quantities of emissions since the rate at which they form may be limited by factors other than the concentration of the precursor gases. For example, secondary ammonium nitrate is not a stable compound. Its equilibrium with gaseous ammonia and nitric acid is strongly influenced by temperature and relative humidity (Watson et al., 1994a). Measurement of these gaseous precursors is needed to determine which gases are in excess and the amounts by which the precursor emissions must be reduced to achieve reductions in particulate nitrate concentrations.

Dust suspended from bare land, roadways, agricultural fields and construction sites is predominantly a primary pollutant, but it does play a role in secondary particle formation (Chow and Watson, 1992; Chow et al., 1994b). Some components of dust, such as ammonium nitrate fertilizer, may volatilize into ammonia and nitric acid gases, thereby contributing to secondary aerosol. Alkaline particles, such as calcium carbonate, may react with nitric and hydrochloric acid gases while on the ground, in the atmosphere, or on filter samples to form coarse particle nitrates and chlorides. Ammonium sulfate fertilization and minerals such as gypsum (calcium sulfate) may be mistaken for secondary sulfates when particle samples are chemically analyzed.

Table 2-4 summarizes source contributions to PM_{10} in several urban/nonurban areas in the U.S. Several of these studies combine receptor and source models for source apportionment, health risk assessment, and control strategy evaluation. The general PM_{10} source types identified are primary geological material, primary motor vehicle exhaust, primary vegetative burning, primary marine aerosol, and primary industrial emissions (e.g., lead smelter, cement plant, steel mill, incinerator). The apportionment of secondary aerosol to emitters of precursors is only attempted in a few studies (e.g., Lewis et al., 1985; Lowenthal et al., 1989; Watson et al., 1994a, 1996a, 1997) with aerosol evolution modeling. Secondary ammonium nitrate and ammonium sulfate reported in Table 2-4 represent the remaining secondary aerosol which is not accounted for by primary emissions. Naming conventions for source types and source sub-types are not consistent among different states, so comparisons among different studies in Table 2-4 are semi-qualitative. Uncertainties in source contribution estimates and model performance measures should be reported to provide a validity assessment of each model application (Watson et al., 1994c), and this reporting has been inconsistent among the different studies.

The sampling sites in Table 2-4 represent a variety of different source characteristics within different regions of Arizona, California, Colorado, Idaho, Illinois, Nevada and Ohio. Several of these are background sites, specifically Estrella Park, Gunnery Range, Pinnacle Peak, and Corona de Tucson, AZ, Anacapa Island, CA, San Nicolas Island, CA, Vandenberg Air Force Base, CA, and Verdi, NV. Definitions of source categories also vary from study to study. In spite of these differences, several features can be observed from the values in this table.

Fugitive dust (geological material) from roads, agriculture and erosion is a major contributor to PM_{10} at nearly all sampling sites, often contributing up to, but not generally more than 50% of the average PM_{10} mass concentration. The average fugitive dust source contribution is highly variable among sampling sites within the same urban areas, as seen by differences between Central Phoenix (33 $\mu g/m^3$) and Scottsdale (25 $\mu g/m^3$) in Arizona. It is also highly variable between seasons, as evidence by the summer and winter contributions at Rubidoux, CA. In general, these studies found that fugitive dust was chemically similar, even though it came from different emitters, so that further apportionment into sub-categories was not possible. An exception was for road sanding in Telluride, CO. Road sand often contains salts that allow it to be distinguished from other fugitive dust sources. It is usually the only exposed fugitive dust source when other sources are covered by snowpack. Dust from some construction activities and cement plants can also be separated from other sources due to enrichments in calcium content of these emissions, as seen in studies at Rubidoux, CA and Rillito, AZ (near cement plants), in Pocatello, ID (near chemical and fertilizer production plants), and Tucson, AZ (where a nearby community center was undergoing renovation).

Primary motor vehicle exhaust contributions account for up to approximately 40% of average PM_{10} at many of the sampling sites. Vehicle exhaust contributions are also variable at different sites within the same non-attainment area. Vegetative biomass burning, which includes agricultural fires, wildfires, prescribed burning, and residential wood combustion, was found to be significant at residential sampling sites such as: Craycroft, Scottsdale, and West Phoenix, AZ; San Jose, Fresno, Bakersfield, and Stockton, CA; Telluride, CO; Sparks, NV; and Mingo, OH. The predominance of these contributions during winter months and the local rather than regional coverage indicates that residential wood combustion was the major sub-category, even though chemical profiles are too similar to separate residential combustion from other vegetative burning sources. For example, Chow et al. (1988) show substantial differences between the residential Sparks, NV, and urban-commercial Reno, NV, burning contributions even though these sites are separated by less than 10 km.

Sites near documented industrial activity show evidence of that activity, but not necessarily from primary particles emitted by point sources. Hayden, AZ, for example, contains a large smelter, but the major smelter contributions appear to arise from fugitive emissions or copper tailings rather than stack emissions. Secondary sulfate contributions at Hayden were low, even though sulfur dioxide emissions from the stack were substantial during the time of the study. Fellows, CA, is in the midst of oilfield facilities that burn crude oil for tertiary oil extraction. The Follansbee, Mingo, Sewage Plant, Steubenville, and Tower sites in Ohio are all close to each other in the Ohio River Valley and show evidence of the widespread steel mill emissions in that area.

Marine aerosol is found, as expected, at coastal sites such as Long Beach, San Nicholas Island, and Anacapa Island, CA, but these contributions are relatively low compared to contributions from manmade sources.

Of great importance are the contributions from secondary ammonium sulfate and ammonium nitrate. These are especially noticeable at sites in California's San Joaquin Valley (Bakersfield, Crows Landing, Fellow, Fresno, Kern Wildlife, and Stockton), in the Los Angeles area, and in the Ohio River Valley. Nitrate was not measured at the Ohio sites, but there was a large portion of unexplained mass in the CMB source apportionments that could be composed in part by ammonium nitrate.

Other aerosol characterization and receptor model source apportionment studies have been performed for $PM_{2.5}$ that could be added to Table 2-4. With the exception of geological material contributions, which are not expected to be significant in $PM_{2.5}$, the general conclusions drawn from this table would not change substantially.

2.4 Particle Properties that Affect Human Exposure and Health

The $PM_{2.5}$ and PM_{10} NAAQS result from an extensive review of scientific studies relating particle concentrations to health, most of them conducted in U.S. cities (U.S. EPA, 1996). Most of these studies show positive and statistically significant relationships between health end-points and different indicators of suspended particles. Many of these indicators are other than $PM_{2.5}$ or PM_{10} mass concentrations. The associations between particle concentrations and health are inconsistent, and more scientific studies are needed to address properties that affect health.

Figure 2-8 shows the fraction of particles with different sizes that deposit in different parts of the human body when particle-laden air is breathed (Phalen et al., 1991; American Conference of Governmental Industrial Hygienists, 1993). Phalen et al. (1991) generated these curves using a model proposed by the National Council on Radiation Protection and Measurements (NCRP), and they are consistent with earlier measurements by Heyder et al. (1986). The International

Standards Organization (ISO) curve is consistent with the mouth-breathing measurements of Swift and Proctor (1982). Most particles larger than 10 µm are removed in the mouth or nose prior to entering the body. Ten to 60% of the particles passing the trachea with aerodynamic diameters less than 10 µm may deposit in the lung where they might cause harm. The lung deposition curve is bimodal, peaking at 20% for ~3 µm particles and at 60% for ~0.03 µm particles.

Swift (1995) notes that high deposition in the nasal area may be related to upper respiratory diseases such as rhinitis, allergy, and sinus infections. The ISO curve is similar to the "ideal inlet" sampling effectiveness that is part of the performance standard for PM_{10} samplers in the United States (U.S. EPA, 1987). These curves show that the amount of particles larger than 2 or 3 µm transmitted through mouth-breathing is significantly larger than the amount transmitted when breathing takes place through the nose.

Epidemiological studies (e.g., Ostro, 1993; Dockery and Pope, 1994; Schwartz, 1994; Kinney et al., 1995; Lippman and Ito, 1995; Lipfert and Wyzga, 1995, 1997; Vedal, 1997; Riveros-Rosas et al., 1997) attempt to determine relationships between ambient concentrations and health indicators, such as hospital admissions, frequencies of respiratory illness, reduced lung capacity, and death. A lower threshold for particle mass concentrations has not been found in these studies (Pope et al., 1995), increases of 0.7% to 1.6% in daily mortality have been observed for each 10 µg/m^3 increase in PM_{10} concentration, regardless of the area studied. Though there are large uncertainties and methodological differences in these studies, many studies show positive relationships between higher particulate concentrations and poorer human health. Pope et al. (1995) identify confounding variables, such as influences from other pollutants, smoking, and changes in weather, but they note that ". . . taken together it is unlikely that such confounding could be consistently acting in all these studies."

While epidemiological studies show relationships, they do not explain how particulate matter damages health. Controlled toxicological studies have shown that specific constituents of suspended particulate matter are associated with specific aggravations to health, but usually at levels far in excess of those found in ambient air (Schlesinger, 1995).

Organic compounds, especially those found in diesel exhaust, have been demonstrated to induce cancer in rats (Klingenberg and Winneke, 1990; Mauderly, 1992). Sulfuric acid has been shown to impair lung clearance (Schlesinger, 1990) and has been considered for designation as a NAAQS (U.S. EPA, 1989). Goyer (1986) shows that several trace metals can suppress the human immune system. The well-established toxic effects of lead resulted in specific NAAQS for this metal and the phase-out of leaded fuels over the past two decades. Crystalline silica, the cause of silicosis in miners, may also have effects at lower concentrations than those found in industrial situations. Different valence states for metals such as iron and chromium may have different toxicities (Bates, 1995). The extrapolation of results obtained from animals to human physiology is not perfect, and effects may be found at lower concentrations in some humans. There is also a dearth of laboratory data on the complex particle mixtures to which humans are actually exposed in ambient air (Schlesinger, 1995).

Kao and Friedlander (1995) speculate that particle mass concentration measurements are merely a surrogate for highly reactive species, such as free radicals, that engender secondary particle formation. These radicals are so short-lived that they can probably never be measured, only inferred from their reaction products (e.g., ozone, nitric acid, nitrates, sulfates, and secondary organic compounds). If such speculation is borne out by health studies, then fine particles, especially the reactive end-products of secondary aerosol formation, would be among the best indicators of adverse health effects from a variety of reactive compounds.

Vedal (1997) reviewed and evaluated nearly 200 scientific publications regarding ambient particles and health and found basic disagreements among different researchers about how epidemiological data should be interpreted. The relationship between health endpoints and particle concentrations appears to reflect a causal relationship, but plausible alternative explanations for the associations can be justified. In particular, changes in meteorology also correspond with changes in pollution and health indicators, thereby potentially confounding statistical associations. Concerns about confounding by such a factor are aggravated by the small size of the estimated particle effects.

Most epidemiological studies estimate ambient particle concentrations from fixed site monitors that do not correlate well with integrated concentrations measured with personal monitors. This can bias the effects estimated from the epidemiological studies in unpredictable ways. It is not certain whether the impact of this misclassification on the particle-associated health findings has been large or small. Siting criteria for the $PM_{2.5}$ monitoring network emphasizes community-oriented monitoring sites to better represent population exposure (Watson et al., 1997).

Vedal (1997) reported that although North American epidemiological studies consistently find positive and significant associations between particles and health, these associations are not as consistent or strong in recent European epidemiological studies. The reasons for these less consistent findings are not known, although differences in particle composition are a possibility.

Vedal (1997) did not find sufficient evidence that acidic particles cause more serious effects than non-acidic particles at typical ambient concentrations. The pathogenicity of minimally acidic ambient particles in western regions of the United States and Canada is clearly not related to the acid aerosol component. Although ultrafine particles (particles with aerodynamic diameters less than 0.1 μm) have been shown to be pathogenic in laboratory studies, significant concentrations have not yet been commonly measured in ambient air.

Compliance measurements are taken at fixed monitoring sites for specified time intervals, usually not less than 24 hours. The air that people breathe depends on where they are, the most common locations being the home, the workplace, the automobile, and the outdoors. Most outdoor human exposure occurs during the daytime, so it is important to understand how particle concentrations differ between day and night. The sampler location, especially its proximity to local sources, can play a large role in its ability to assess human exposure.

Comparisons of outdoor particle mass concentrations with corresponding measurements indoors and with personal exposure monitors carried by test subjects generally show poor correlations (Sexton et al., 1984; Morandi et al.,1988; Lioy et al, 1990; Pellizzari et al., 1993; Suh et al., 1993). The correspondence between these three types of samples is much better for some chemical species, such as sulfate (Suh et al., 1993). Particles from smoking, cooking, house dust, and other indoor emissions often constitute the remainder of indoor concentrations.

The lack of correlation between indoor and outdoor measurements, therefore, does not mean that outdoor concentrations are unimportant. While residents can control indoor emissions through personal actions such as using filtered vacuum cleaners and exhausting cooking emissions, there is little that they can do to prevent the incursion of pollution from outdoor air. Smaller particles, such as $PM_{2.5}$, are more likely to penetrate indoors than are the coarse particles, which are more likely to deposit within the cracks and seams where air penetrates. Coarse particles also deposit to surfaces more rapidly due to gravitational settling in the stilled air of most indoor environments.

Most of the evidence relating ambient measurements of suspended particles taken in compliance networks to personal exposures shows that: (1) ambient concentrations, especially those for $PM_{2.5}$ particles, constitute a major fraction of the particles to which humans are exposed; and (2) ambient levels generally represent a lower bound on the concentrations to which people are commonly exposed.

The chemically speciated data base resulting from the $PM_{2.5}$ speciation monitoring network will allow additional associations between health and particles to be delineated. In addition to the first-order chemical speciation of elements, ions, and carbon, other aerosol properties such as ultrafine particles, organics (especially polycyclic aromatic hydrocarbons [PAHs]), and single particle characterization should be sought to establish their causality with health indicators.

Table 2-1
Examples of Organic Compounds Found in Different Emission Sources and in Ambient Air

Species	Predominant Sources	Particle-Gas Phase Distribution
PAH, for example		
naphthalene	Motor vehicles, wood smoke	Gas Phase
methylnaphthalenes	Motor vehicles, wood smoke	Gas Phase
dimethylnaphthalenes	Motor vehicles, wood smoke	Gas Phase
biphenyl	Motor vehicles, wood smoke	Gas Phase
acenaphthylene	Motor vehicles, wood smoke	Gas Phase
acenaphthene	Motor vehicles, wood smoke	Gas Phase
fluorene	Motor vehicles, wood smoke	Gas Phase
phenanthrene	Motor vehicles, wood smoke	Particle-Gas Phase
anthracene	Motor vehicles, wood smoke	Particle-Gas Phase
fluoranthene	Motor vehicles, wood smoke	Particle-Gas Phase
pyrene	Motor vehicles, wood smoke	Particle-Gas Phase
retene	Wood smoke -softwood	Particle-Gas Phase
benzo[b]naphtho[2,1]thiophene	Motor vehicles	Particle Phase
benz[a]anthracene	Motor vehicles, wood smoke	Particle Phase
chrysene	Motor vehicles, wood smoke	Particle Phase
benzo[b+j+k]fluoranthene	Motor vehicles, wood smoke	Particle Phase
benzo[e]pyrene	Motor vehicles, wood smoke	Particle Phase
benzo[a]pyrene	Motor vehicles, wood smoke	Particle Phase
indene[123-cd]pyrene	Motor vehicles, wood smoke	Particle Phase
dibenzo[ah+ac]anthracene	Motor vehicles, wood smoke	Particle Phase
benzo[ghi]perylene	Motor vehicles, wood smoke	Particle Phase
coronene	Motor vehicles, wood smoke	Particle Phase
Hopanes and Sterenes		
Cholestanes	Motor vehicles	Particle Phase
Trisnorhopanes	Motor vehicles	Particle Phase
Norhopanes	Motor vehicles	Particle Phase
Hopanes	Motor vehicles	Particle Phase
Guaiacols, for example		
4-methylguaiacol	Wood smoke	Gas Phase
4-allylguaiacol	Wood smoke	Particle-Gas Phase
isouegenol	Wood smoke	Particle-Gas Phase
Acetovanillone	Wood smoke	Particle Phase
Syringols, for example		
Syringol	Wood smoke, mostly hardwood	Particle-Gas Phase
4-methylsyringol	Wood smoke, mostly hardwood	Particle-Gas Phase
Syringaldehyde	Wood smoke, mostly hardwood	Particle Phase
Lactons, for example		
Caprolactone	Meat cooking	Gas Phase
Decanolactone	Meat cooking	Particle-Gas Phase
Undecanoic-G-Lactone	Meat cooking	Particle-Gas Phase
Sterols, for example		
Cholesterol	Meat cooking	Particle Phase
Sitosterol	Meat cooking, wood smoke	Particle Phase

Table 2-2

Average PM$_{10}$ Composition (μg/m^3) in Selected Urban and Non-Urban U.S. Areas

Study	Site	Sampling StartDate	Sampling EndDate	Integration Time (h)	# in Avg	Mass	Cl-	NO3-	SO4=	NH4+	EC	OC	Al	Si	P
Bay Area	San Carlos St., CA	12/16/91	02/24/92	12 day	9	53.2	0.21	11.78	2.14	4.01	6.20	14.16	0.81	2.98	0.009
Bay Area	San Carlos St., CA	12/16/91	02/24/92	12 night	13	66.7	0.99	8.29	1.94	2.89	9.51	22.65	0.65	2.39	0.009
Bay Area	Fourth St., CA	12/16/91	02/24/92	12 day	9	56.0	0.24	12.02	2.52	4.20	6.98	14.30	0.93	2.93	0.006
Bay Area	Fourth St., CA	12/16/91	02/24/92	12 night	13	69.3	1.06	9.76	2.16	3.44	10.19	22.75	0.78	2.55	0.005
AUSPEX1	Point Reyes, CA	07/13/90	08/24/90	0000-2400	13	7.27	2.28	0.47	1.98	0.25	0.48	1.24	0.0030	0.0180	0.0060
AUSPEX1	Altamont Pass, CA	07/13/90	08/24/90	0000-2400	14	23.29	0.43	1.30	2.97	0.82	2.04	6.83	1.1470	3.5340	0.0120
AUSPEX1	Pacheco Pass, CA	07/13/90	08/24/90	0000-2400	14	16.17	0.35	1.47	2.86	0.78	1.85	6.45	0.5520	2.0660	0.0000
AUSPEX1	Crows Landing, CA	07/13/90	08/24/90	0000-2400	14	69.87	0.20	1.79	2.92	0.91	2.20	8.89	6.9720	15.7200	0.0100
AUSPEX1	Academy, CA	07/13/90	08/24/90	0000-2400	14	35.92	0.13	0.99	2.65	0.93	1.60	8.58	2.2180	6.1430	0.0300
AUSPEX1	Buttonwillow, CA	07/13/90	08/24/90	0000-2400	14	53.70	0.20	1.53	2.98	0.80	2.32	8.04	3.6970	11.2850	0.0170
AUSPEX1	Edison, CA	07/13/90	08/24/90	0000-2400	14	52.46	0.09	1.60	3.29	1.07	2.97	10.34	2.6840	6.7080	0.0500
AUSPEX1	Caliente, CA	07/13/90	08/24/90	0000-2400	14	33.49	0.04	0.96	2.91	1.01	3.72	8.17	1.5070	4.0360	0.0090
AUSPEX1	Sequoia, CA	07/13/90	08/24/90	0000-2400	14	21.14	0.00	0.69	1.89	0.78	1.96	5.21	1.1350	2.9230	0.0020
AUSPEX1	Yosemite, CA	07/13/90	08/24/90	0000-2400	14	23.66	0.08	0.32	1.90	0.65	1.75	10.44	1.1450	2.7020	0.0020
IMS95	Corcoran, CA	11/06/95	11/14/95	0000-2400	9	120.97	0.21	17.28	2.90	4.56	3.05	15.79	6.1441	18.7136	0.1140
IMS95	Chowchilla, CA	12/25/95	01/06/96	0000-2400	5	42.58	0.29	16.14	2.16	5.49	2.37	5.95	0.3114	0.8066	0.0167
IMS95	Kern Wildlife Refuge, CA	12/25/95	01/06/96	0000-2400	9	38.52	0.29	15.65	2.11	5.35	1.92	4.62	0.3163	0.9545	0.0112
IMS95	Fresno, CA	12/25/95	01/06/96	0000-2400	9	76.88	0.65	15.08	2.42	5.24	8.06	22.67	0.5658	1.6318	0.0187
IMS95	Bakersfield, CA	12/25/95	01/06/96	0000-2400	9	64.47	0.65	16.42	2.82	5.84	6.79	15.08	0.8742	2.4593	0.0117
Calexico	Calexico, CA	09/03/92	08/23/93	0000-2400	55	62.50	1.19	2.25	2.75	1.12	2.50	10.09	3.8729	11.3956	0.0269
Calexico	Mexicali, CA	09/03/92	08/23/93	0000-2400	48	130.79	3.48	2.69	4.40	1.58	3.78	24.39	7.0710	20.6537	0.0210
Calexico	Calexico, CA	03/13/92	08/28/92	0000-2400	25	43.11	0.74	1.53	2.97	0.87	1.39	7.91	2.6815	7.9101	0.0205
Calexico	Mexicali, CA	03/13/92	08/28/92	0000-2400	25	82.91	1.71	1.76	3.53	0.81	1.86	14.04	5.2318	15.3345	0.0282
Calexico	Calexico, CA	09/03/92	08/23/93	0000-2400	48	57.24	N/A	N/A	N/A	N/A	N/A	N/A	4.0190	11.7056	0.0170
Calexico	Mexicali, CA	09/03/92	08/23/93	0000-2400	44	125.56	N/A	N/A	N/A	N/A	N/A	N/A	9.2806	26.9183	0.0364
Calexico	Calexico, CA	03/13/92	08/28/92	0000-2400	27	49.22	N/A	N/A	N/A	N/A	N/A	N/A	3.1140	9.1519	0.0165
Calexico	Mexicali, CA	03/13/92	08/28/92	0000-2400	25	92.12	N/A	N/A	N/A	N/A	N/A	N/A	6.0240	17.7068	0.0308
Calexico	Calexico, CA	12/11/92	01/07/93	0000-2400	27	39.11	1.01	3.56	2.15	1.66	2.90	8.65	1.4523	4.2032	0.0052
Calexico	Mexicali, CA	12/11/92	01/07/93	0000-2400	18	145.73	5.18	4.38	4.78	3.16	8.10	37.32	5.3517	15.5319	0.0011
Calexico	Calexico, CA	12/21/92	01/07/93	0000-2400	22	45.95	1.64	3.22	1.96	1.74	4.32	10.02	1.8772	5.4471	0.0070
Calexico	Mexicali, CA	12/21/92	01/07/93	0000-2400	14	137.04	5.53	4.09	4.06	3.32	8.85	33.55	5.7432	16.7210	0.0043

Table 2-2 (continued)
Average PM_{10} Composition ($\mu g/m^3$) in Selected Urban and Non-Urban U.S. Areas

Study	Site	V	Cr	Mn	Fe	Ni	Cu	Zn	Se	Br	Rb	Sr	Zr	Ba	Pb
Bay Area	San Carlos St., CA	0.004	0.001	0.012	0.785	0.002	0.020	0.053	0.003	0.010	0.001	0.007	0.002	#N/A	0.025
Bay Area	San Carlos St., CA	0.010	0.003	0.012	0.723	0.002	0.035	0.060	0.003	0.011	0.002	0.010	0.001	#N/A	0.034
Bay Area	Fourth St., CA	0.004	0.003	0.014	0.824	0.004	0.020	0.059	0.003	0.011	0.001	0.016	0.002	#N/A	0.031
Bay Area	Fourth St., CA	0.004	0.002	0.014	0.863	0.004	0.030	0.073	0.003	0.012	0.001	0.028	0.006	#N/A	0.042
AUSPEX1	Point Reyes, CA	0.0020	0.0010	0.0010	0.0140	0.0010	0.0040	#N/A	0.0010	0.0050	0.0000	0.0010	0.0010	0.0290	0.0020
AUSPEX1	Altamont Pass, CA	0.0040	0.0030	0.0150	0.6170	0.0020	0.0040	#N/A	0.0020	0.0060	0.0010	0.0050	0.0020	0.0330	0.0120
AUSPEX1	Pacheco Pass, CA	0.0020	0.0010	0.0090	0.3640	0.0030	0.0030	#N/A	0.0000	0.0030	0.0010	0.0020	0.0010	0.0350	0.0030
AUSPEX1	Crows Landing, CA	0.0060	0.0050	0.0580	3.1610	0.0060	0.0080	#N/A	0.0020	0.0070	0.0070	0.0140	0.0060	0.0900	0.0120
AUSPEX1	Academy, CA	0.0030	0.0010	0.0260	1.1870	0.0030	0.0190	#N/A	0.0010	0.0080	0.0030	0.0070	0.0020	0.0510	0.0080
AUSPEX1	Buttonwillow, CA	0.0070	0.0020	0.0340	1.9290	0.0060	0.0050	#N/A	0.0010	0.0090	0.0050	0.0150	0.0040	0.0550	0.0080
AUSPEX1	Edison, CA	0.0030	0.0010	0.0360	2.1400	0.0040	0.0110	#N/A	0.0010	0.0100	0.0050	0.0130	0.0030	0.0680	0.0100
AUSPEX1	Caliente, CA	0.0020	0.0010	0.0200	1.1320	0.0020	0.0100	#N/A	0.0010	0.0100	0.0020	0.0060	0.0010	0.0630	0.0090
AUSPEX1	Sequoia, CA	0.0010	0.0010	0.0140	0.5270	0.0010	0.0130	#N/A	0.0010	0.0060	0.0010	0.0030	0.0010	0.0330	0.0040
AUSPEX1	Yosemite, CA	0.0020	0.0000	0.0130	0.5670	0.0010	0.0020	#N/A	0.0000	0.0040	0.0010	0.0030	0.0010	0.0540	0.0020
IMS95	Corcoran, CA	0.0082	0.0058	0.0646	3.9927	0.0024	0.0144	0.0583	0.0016	0.0118	0.0099	0.0268	0.0045	0.0437	0.0143
IMS95	Chowchilla, CA	0.0004	0.0003	0.0026	0.1657	0.0011	0.0439	0.1952	0.0012	0.0052	0.0004	0.0011	0.0004	0.0139	0.0153
IMS95	Kern Wildlife Refuge, CA	0.0008	0.0004	0.0038	0.1838	0.0021	0.0061	0.0427	0.0013	0.0045	0.0004	0.0018	0.0007	0.0090	0.0067
IMS95	Fresno, CA	0.0011	0.0011	0.0070	0.5269	0.0010	0.0159	0.0544	0.0009	0.0104	0.0010	0.0050	0.0010	0.0285	0.0188
IMS95	Bakersfield, CA	0.0019	0.0012	0.0099	0.7111	0.0026	0.0317	0.0628	0.0010	0.0119	0.0013	0.0042	0.0011	0.0324	0.0184
Calexico	Calexico, CA	0.0096	0.0027	0.0301	1.6123	0.0018	0.0110	0.0400	0.0017	0.0127	0.0049	0.0217	0.0060	0.0522	0.0385
Calexico	Mexicali, CA	0.0239	0.0068	0.0643	3.5436	0.0040	0.0320	0.0988	0.0039	0.0239	0.0109	0.0421	0.0116	0.0873	0.0968
Calexico	Calexico, CA	0.0097	0.0018	0.0203	1.0303	0.0017	0.0079	0.0254	0.0017	0.0106	0.0034	0.0130	0.0039	0.0251	0.0193
Calexico	Mexicali, CA	0.0213	0.0041	0.0427	2.2895	0.0034	0.0232	0.0722	0.0028	0.0163	0.0072	0.0268	0.0080	0.0408	0.0603
Calexico	Calexico, CA	0.0124	0.0029	0.0273	1.4707	0.0025	0.0109	0.0383	0.0018	0.0128	0.0044	0.0180	0.0049	0.0331	0.0348
Calexico	Mexicali, CA	0.0215	0.0059	0.0627	3.5847	0.0034	0.0298	0.1146	0.0037	0.0212	0.0108	0.0413	0.0113	0.0722	0.0938
Calexico	Calexico, CA	0.0105	0.0017	0.0236	1.1789	0.0019	0.0089	0.0389	0.0015	0.0089	0.0039	0.0142	0.0040	0.0325	0.0256
Calexico	Mexicali, CA	0.0212	0.0040	0.0458	2.4041	0.0034	0.0286	0.0799	0.0027	0.0151	0.0073	0.0282	0.0093	0.0495	0.0667
Calexico	Calexico, CA	0.0022	0.0009	0.0109	0.6051	0.0008	0.0076	0.0318	0.0019	0.0083	0.0017	0.0180	0.0023	0.0537	0.0288
Calexico	Mexicali, CA	0.0138	0.0061	0.0466	2.6442	0.0028	0.0336	0.1102	0.0047	0.0368	0.0083	0.0476	0.0085	0.1262	0.1208
Calexico	Calexico, CA	0.0024	0.0016	0.0137	0.7522	0.0010	0.0090	0.0397	0.0016	0.0114	0.0023	0.0175	0.0030	0.0640	0.0344
Calexico	Mexicali, CA	0.0129	0.0054	0.0468	2.6078	0.0026	0.0309	0.1115	0.0039	0.0341	0.0082	0.0422	0.0083	0.1167	0.1089

Table 2-2 (continued)
Average PM$_{10}$ Composition (μg/m^3) in Selected Urban and Non-Urban U.S. Areas

Study	Site	Sampling StartDate	Sampling EndDate	Integration Time (h)	# in Avg	Mass	Cl-	NO3-	SO4=	NH4+	EC	OC	Al	Si	P
SCAQS4	Burbank, CA	06/19/87	09/03/87	0000-2400	11	72.30	0.82	10.64	10.65	5.01	3.09	12.76	0.8264	2.1932	0.0729
SCAQS4	Downtown Los Angeles, CA	06/19/87	09/03/87	0000-2400	11	67.40	0.96	9.47	11.28	4.61	3.19	11.61	0.7578	2.0397	0.1871
SCAQS4	Hawthorne, CA	06/19/87	09/03/87	0000-2400	11	45.90	1.27	4.97	11.17	3.76	0.98	4.66	0.4854	1.2927	0.0255
SCAQS4	Long Beach, CA	06/19/87	09/03/87	0000-2400	11	46.10	0.81	5.15	8.74	2.84	1.47	5.09	0.7095	1.8080	0.0460
SCAQS4	Anaheim, CA	06/19/87	09/03/87	0000-2400	11	51.30	0.94	7.16	8.05	3.19	1.70	7.16	0.7012	1.9228	0.0514
SCAQS4	Rubidoux, CA	06/19/87	09/03/87	0000-2400	11	120.60	0.88	28.84	8.81	8.60	3.15	14.66	2.1214	5.2886	0.3606
SCAQS4	San Nicolas Island, CA	06/19/87	09/03/87	0000-2400	11	17.40	1.64	1.58	3.57	0.85	0.16	1.53	0.1324	0.3376	0.0027
SCAQS4	Azusa, CA	06/19/87	09/03/87	0000-2400	11	92.10	0.64	10.62	9.91	4.85	3.33	13.46	2.2705	5.7213	0.1046
SCAQS4	Claremont, CA	06/19/87	09/03/87	0000-2400	11	70.00	0.39	10.14	8.15	3.62	2.69	12.88	1.2318	3.1104	0.0923
SCAQS4	Burbank, CA	11/11/87	12/11/87	0000-2400	6	94.80	0.76	25.74	4.52	8.90	7.38	22.12	0.9514	2.2984	0.0908
SCAQS4	Downtown Los Angeles, CA	11/11/87	12/11/87	0000-2400	6	98.70	0.81	27.50	5.39	8.67	8.49	23.35	0.8470	2.1624	0.0988
SCAQS4	Hawthorne, CA	11/11/87	12/11/87	0000-2400	6	85.10	1.84	21.19	5.97	7.07	6.92	17.64	0.8168	2.0123	0.0756
SCAQS4	Long Beach, CA	11/11/87	12/11/87	0000-2400	6	96.10	1.47	24.75	5.40	7.33	7.27	23.84	1.0222	2.4227	0.1123
SCAQS4	Anaheim, CA	11/11/87	12/11/87	0000-2400	6	104.00	1.51	35.29	5.06	10.34	6.76	18.46	1.0976	2.8801	0.0862
SCAQS4	Riverside, CA	11/11/87	12/11/87	0000-2400	6	112.00	0.89	29.65	3.68	6.36	6.86	17.32	2.0132	5.0600	0.1121
Rubidoux	Rubidoux, CA	10/01/88	04/01/89	0000-2400	61	87.45	0.75	20.23	5.41	5.38	4.38	10.31	4.21	11.54	0.095
Rubidoux	Magnolia, CA	10/01/88	04/01/89	0000-2400	61	66.29	0.58	16.96	4.61	5.25	4.19	9.07	2.90	7.85	0.054
Rubidoux	Riverside, CA	10/01/88	04/01/89	0000-2400	61	63.43	0.56	17.83	4.56	5.65	3.22	7.48	3.34	8.52	0.046
Las Vegas	Bemis, NV	01/03/95	01/28/96			28.35	0.12	0.61	1.04	0.21	1.50	3.64	1.0714	4.4414	0.0074
Las Vegas	East Charleston, NV	01/03/95	01/28/96			33.31	0.21	1.00	1.64	0.28	3.82	7.40	1.2555	5.1862	0.0206
Phoenix PM10	West Phoenix, AZ	09/25/89	01/21/90	0000-2400	11	68.74	0.53	4.99	2.03	1.96	10.19	18.28	2.8170	7.6646	0.0619
Phoenix PM10	Central Phoenix, AZ	09/25/89	01/21/90	0000-2400	9	64.05	0.85	4.08	1.46	1.63	6.64	11.32	2.8702	8.1926	0.0558
Phoenix PM10	South Scottsdale, AZ	09/25/89	01/21/90	0000-2400	11	54.61	0.36	4.30	1.63	1.60	8.15	14.05	2.3234	6.4695	0.0450
Phoenix PM10	Estrella Park, AZ	09/25/89	01/21/90	0000-2400	10	54.75	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	4.0671	8.2164	0.0715
Phoenix PM10	Gunnery Range, AZ	09/25/89	01/21/90	0000-2400	10	26.84	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	2.2031	5.7325	0.0335
Phoenix PM10	Pinnacle Peak, AZ	09/25/89	01/21/90	0000-2400	11	11.77	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	0.7114	1.9145	0.0120
Pilot Tucson PM	Downtown Tucson, AZ	09/29/89	01/23/90	0000-2400	7	47.96	0.13	1.01	1.77	0.62	2.74	7.48	2.2800	6.8700	0.0370
Pilot Tucson PM	Orange Grove, AZ	09/29/89	01/23/90	0000-2400	7	34.20	0.09	1.36	1.54	0.74	3.44	6.24	2.2800	6.0820	0.0370
Pilot Tucson PM	22nd and Craycroft, AZ	09/29/89	01/23/90	0000-2400	7	23.39	0.07	1.01	1.41	0.69	2.13	4.01	1.4500	3.5500	0.0200
Pilot Tucson PM	Corona de Tucson, AZ	09/29/89	01/23/90	0000-2400	6	19.08	N/A	N/A	N/A	N/A	N/A	N/A	1.2800	3.5800	0.0028

Table 2-2 (continued)
Average PM_{10} Composition ($\mu g/m^3$) in Selected Urban and Non-Urban U.S. Areas

Study	Site	V	Cr	Mn	Fe	Ni	Cu	Zn	Se	Br	Rb	Sr	Zr	Ba	Pb
SCAQS4	Burbank, CA	0.0052	0.0247	0.0282	0.8353	0.0045	0.0071	0.0445	0.0083	0.0092	#N/A	0.0180	#N/A	0.0734	0.0784
SCAQS4	Downtown Los Angeles, CA	0.0052	0.0232	0.0327	0.8357	0.0046	0.0224	0.1138	0.0081	0.0163	#N/A	0.0179	#N/A	0.0701	0.0844
SCAQS4	Hawthorne, CA	0.0058	0.0217	0.0164	0.3789	0.0045	0.0305	0.0365	0.0083	0.0090	#N/A	0.0177	#N/A	0.0228	0.0437
SCAQS4	Long Beach, CA	0.0068	0.0209	0.0213	0.5554	0.0050	0.0061	0.0410	0.0091	0.0101	#N/A	0.0212	#N/A	0.0343	0.0614
SCAQS4	Anaheim, CA	0.0051	0.0196	0.0236	0.5989	0.0047	0.0103	0.0248	0.0084	0.0094	#N/A	0.0187	#N/A	0.0420	0.0504
SCAQS4	Rubidoux, CA	0.0084	0.0241	0.0607	1.8098	0.0048	0.0073	0.0242	0.0089	0.0093	#N/A	0.0184	#N/A	0.0726	0.0632
SCAQS4	San Nicolas Island, CA	0.0041	0.0195	0.0120	0.0755	0.0062	0.0169	0.0118	0.0078	0.0092	#N/A	0.0180	#N/A	0.0052	0.0330
SCAQS4	Azusa, CA	0.0075	0.0196	0.0792	1.8409	0.0046	0.0136	0.1680	0.0087	0.0092	#N/A	0.0184	#N/A	0.1375	0.0811
SCAQS4	Claremont, CA	0.0060	0.0186	0.0412	1.0444	0.0050	0.0159	0.0848	0.0089	0.0100	#N/A	0.0199	#N/A	0.0634	0.0858
SCAQS4	Burbank, CA	0.0062	0.0384	0.0710	2.0477	0.0057	0.1489	0.2266	0.0139	0.0728	#N/A	0.0264	#N/A	0.1252	0.2644
SCAQS4	Downtown Los Angeles, CA	0.0092	0.0420	0.0633	2.1918	0.0045	0.1783	0.2934	0.0096	0.0715	#N/A	0.0238	#N/A	0.1267	0.2512
SCAQS4	Hawthorne, CA	0.0129	0.0408	0.0632	1.7369	0.0216	0.2296	0.3178	0.0121	0.0816	#N/A	0.0245	#N/A	0.1003	0.2592
SCAQS4	Long Beach, CA	0.0125	0.0356	0.0752	1.9580	0.0093	0.1261	0.2953	0.0090	0.0868	#N/A	0.0247	#N/A	0.0984	0.2860
SCAQS4	Anaheim, CA	0.0116	0.0334	0.0627	1.9107	0.0114	0.0628	0.1788	0.0099	0.0707	#N/A	0.0324	#N/A	0.0850	0.2346
SCAQS4	Riverside, CA	0.0099	0.0323	0.0985	2.9547	0.0049	0.0593	0.1269	0.0102	0.0670	#N/A	0.0266	#N/A	0.0820	0.1961
Rubidoux	Rubidoux, CA	0.016	0.014	0.067	2.53	0.0077	0.086	0.10	0.0016	0.020	0.0068	0.031	0.0065	0.074	0.071
Rubidoux	Magnolia, CA	0.011	0.010	0.046	1.70	0.0059	0.025	0.059	0.0014	0.018	0.0039	0.019	0.0035	0.060	0.069
Rubidoux	Riverside, CA	0.011	0.0094	0.042	1.73	0.0050	0.029	0.059	0.0018	0.014	0.0047	0.019	0.0037	0.050	0.049
Las Vegas	Bemis, NV	0.0019	0.0082	0.0153	0.5974	0.0053	0.0065	0.0215	0.0002	0.0024	0.0014	0.0279	0.0020	0.0209	0.0045
Las Vegas	East Charleston, NV	0.0012	0.0014	0.0165	0.9358	0.0007	0.0128	0.0353	0.0002	0.0043	0.0018	0.0267	0.0027	0.0565	0.0105
Phoenix PM10	West Phoenix, AZ	0.0090	0.0119	0.0686	1.5946	0.0067	0.0277	0.1218	0.0009	0.0149	0.0035	0.0190	0.0071	0.0499	0.0730
Phoenix PM10	Central Phoenix, AZ	0.0090	0.0129	0.0540	1.5745	0.0074	0.0460	0.0928	0.0009	0.0158	0.0039	0.0188	0.0067	0.0492	0.0656
Phoenix PM10	South Scottsdale, AZ	0.0074	0.0083	0.0398	1.2555	0.0036	0.0340	0.0543	0.0011	0.0099	0.0033	0.0151	0.0041	0.0437	0.0469
Phoenix PM10	Estrella Park, AZ	0.0077	0.0081	0.0404	1.3745	0.0030	0.0275	0.1708	0.0014	0.0073	0.0037	0.0186	0.0048	0.0343	0.0308
Phoenix PM10	Gunnery Range, AZ	0.0057	0.0044	0.0231	0.8854	0.0024	0.0073	0.0137	0.0006	0.0037	0.0018	0.0100	0.0034	0.0262	0.0152
Phoenix PM10	Pinnacle Peak, AZ	0.0022	0.0019	0.0088	0.2862	0.0007	0.0067	0.0075	0.0006	0.0026	0.0007	0.0034	0.0010	0.0137	0.0078
Pilot Tucson PM	Downtown Tucson, AZ	0.0081	0.0074	0.0400	1.0480	0.0024	0.0630	0.0540	0.0016	0.0096	0.0041	0.0270	0.0035	0.0420	0.0470
Pilot Tucson PM	Orange Grove, AZ	0.0039	0.0050	0.0320	0.7400	0.0016	0.0230	0.0260	0.0015	0.0068	0.0030	0.0074	0.0019	0.0310	0.0310
Pilot Tucson PM	22nd and Craycroft, AZ	0.0030	0.0028	0.0190	0.4600	0.0011	0.0200	0.0200	0.0012	0.0050	0.0018	0.0054	0.0014	0.0220	0.0250
Pilot Tucson PM	Corona de Tucson, AZ	0.0028	0.0022	0.0150	0.4700	0.0008	0.0120	0.0099	0.0004	0.0019	0.0019	0.0045	0.0018	<0.016a	0.0072

Table 2-2 (continued)
Average PM_{10} Composition ($\mu g/m^3$) in Selected Urban and Non-Urban U.S. Areas

Study	Site	Sampling StartDate	Sampling EndDate	Integration Time (h)	# in Avg	Mass	Cl-	NO3-	SO4=	NH4+	EC	OC	Al	Si	P
Robbins	Alsip, IL	10/12/95	09/30/96	0000-2400	16	30.12	NA	4.19	5.45	#N/A	1.93	4.26	0.3208	1.2312	0.0047
Robbins	Breman, IL	10/12/95	09/30/96	0000-2400	17	33.34	NA	4.28	5.89	#N/A	2.05	5.31	0.3800	1.4534	0.0114
Robbins	Meadow Lane, IL	10/12/95	09/30/96	0000-2400	17	30.52	NA	4.39	5.27	#N/A	1.76	4.82	0.4200	1.4345	0.0051
Robbins	Eisenhower, IL	10/12/95	09/30/96	0000-2400	17	32.37	NA	4.06	5.66	#N/A	2.19	4.59	0.4469	1.5690	0.0068

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[a] Chow et al. (1996b).

[b] Chow et al. (1996a).

[c] Chow and Egami (1977).

[d] Chow and Watson (1997b).

[e] Chow et al. (1994a).

[f] Chow et al. (1992b).

[g] Chow and Watson (1997a).

[h] Chow et al. (1991).

[i] Chow et al. (1992c).

[j] Watson et al. (1997d)

Table 2-2 (continued)
Average PM_{10} Composition ($\mu g/m^3$) in Selected Urban and Non-Urban U.S. Areas

Study	Site	V	Cr	Mn	Fe	Ni	Cu	Zn	Se	Br	Rb	Sr	Zr	Ba	Pb
Robbins	Alsip, IL	0.0021	0.0035	0.0163	0.5606	0.0016	0.0259	0.0798	0.0017	0.0054	0.0007	0.0024	0.0013	0.0195	0.0222
Robbins	Breman, IL	0.0028	0.0035	0.0258	0.7054	0.0016	0.0099	0.1108	0.0020	0.0060	0.0009	0.0028	0.0015	0.0255	0.0292
Robbins	Meadow Lane, IL	0.0022	0.0028	0.0189	0.6251	0.0024	0.0096	0.0848	0.0018	0.0055	0.0010	0.0026	0.0156	0.0198	0.0245
Robbins	Eisenhower, IL	0.0027	0.0036	0.0220	0.7407	0.0023	0.0127	0.0901	0.0019	0.0058	0.0009	0.0030	0.0016	0.0288	0.0251

Table 2-3

Average $PM_{2.5}$ Composition ($\mu g/m^3$) in Selected Urban and Non-Urban U.S. Areas

Study	Site	Sampling StartDate	Sampling EndDate	Integration Time (h)	# in Avg	Mass	Cl-	NO3-	SO4=	NH4+	EC	OC	Al	Si
AUSPEX1	Point Reyes, CA	07/13/90	08/24/90	0000-2400	13	2.69	0.46	0.11	1.11	0.23	0.36	1.53	0.0160	0.0100
AUSPEX1	Altamont Pass, CA	07/13/90	08/24/90	0000-2400	14	11.01	0.05	0.24	2.33	0.84	2.61	4.85	0.0760	0.2790
AUSPEX1	Pacheco Pass, CA	07/13/90	08/24/90	0000-2400	13	9.54	0.08	0.47	2.15	0.65	1.02	3.19	0.2370	0.2520
AUSPEX1	Crows Landing, CA	07/13/90	08/24/90	0000-2400	14	29.93	0.09	0.80	3.08	0.82	1.76	7.40	1.3900	3.3920
AUSPEX1	Academy, CA	07/13/90	08/24/90	0000-2400	14	16.95	0.04	0.35	2.45	0.94	1.43	5.95	0.6310	1.0650
AUSPEX1	Buttonwillow, CA	07/13/90	08/24/90	0000-2400	14	18.74	0.07	0.50	2.46	0.76	1.86	6.36	0.4690	1.4540
AUSPEX1	Edison, CA	07/13/90	08/24/90	0000-2400	14	49.65	0.14	1.49	3.33	1.15	2.95	10.02	1.8240	5.0200
AUSPEX1	Caliente, CA	07/13/90	08/24/90	0000-2400	14	19.98	0.02	0.40	2.97	0.98	3.33	7.39	0.4160	1.1800
AUSPEX1	Sequoia, CA	07/13/90	08/24/90	0000-2400	14	10.74	0.01	0.24	2.01	0.71	1.64	5.32	0.1210	0.2010
AUSPEX1	Yosemite, CA	07/13/90	08/24/90	0000-2400	14	15.97	0.06	0.23	1.83	0.49	1.87	12.07	0.1590	0.3910
IMS95	Bakersfield, CA	12/25/95	01/06/96	0000-2400	9	48.93	0.47	14.09	2.46	4.78	5.29	13.23	0.0430	0.1345
IMS95	Fresno, CA	12/25/95	01/06/96	0000-2400	9	61.58	0.49	15.02	2.01	4.28	7.39	20.85	0.0284	0.0897
IMS95	Kern Wildlife Refuge, CA	12/25/95	01/06/96	0000-2400	9	29.82	0.14	14.87	1.64	4.09	1.62	3.48	0.0383	0.1201
IMS95	Chowchilla, CA	12/25/95	01/06/96	0000-2400	5	32.15	0.23	14.04	1.86	4.55	2.04	5.06	0.0219	0.0559
SCAQS4	Burbank, CA	06/19/87	09/03/87	0000-2400	11	42.6	0.21	5.11	8.72	4.30	2.21	9.13	0.0252	0.0449
SCAQS4	Downtown Los Angeles, CA	06/19/87	09/03/87	0000-2400	11	41.1	0.21	4.34	9.41	4.28	2.37	8.27	0.0351	0.0515
SCAQS4	Hawthorne, CA	06/19/87	09/03/87	0000-2400	11	30.5	0.27	1.25	9.67	3.59	0.70	3.35	0.0299	0.0505
SCAQS4	Long Beach, CA	06/19/87	09/03/87	0000-2400	11	25.4	0.15	1.44	7.42	3.02	0.99	3.35	0.0836	0.1526
SCAQS4	Anaheim, CA	06/19/87	09/03/87	0000-2400	11	26.8	0.11	2.36	6.49	2.40	1.20	4.72	0.0351	0.0338
SCAQS4	Rubidoux, CA	06/19/87	09/03/87	0000-2400	11	63.9	0.35	21.19	7.13	8.73	1.73	8.47	0.1329	0.2863
SCAQS4	San Nicolas Island, CA	06/19/87	09/03/87	0000-2400	11	9.7	0.39	0.46	2.77	0.68	0.10	0.81	0.0287	0.0482
SCAQS4	Azusa, CA	06/19/87	09/03/87	0000-2400	11	47.1	0.16	5.09	8.28	4.21	2.64	9.53	0.1874	0.4829
SCAQS4	Claremont, CA	06/19/87	09/03/87	0000-2400	11	41.0	0.12	4.89	6.79	3.71	1.92	9.59	0.0749	0.1785
SCAQS4	Burbank, CA	11/11/87	12/11/87	0000-2400	6	78.3	0.58	22.02	3.76	6.52	6.32	19.55	0.1493	0.3459
SCAQS4	Downtown Los Angeles, CA	11/11/87	12/11/87	0000-2400	6	90.2	0.54	22.64	4.38	6.80	7.28	18.46	0.2502	0.5203
SCAQS4	Hawthorne, CA	11/11/87	12/11/87	0000-2400	6	68.9	1.09	16.57	4.93	5.53	5.81	14.00	0.1633	0.3529
SCAQS4	Long Beach, CA	11/11/87	12/11/87	0000-2400	6	72.7	0.92	19.36	4.42	7.18	6.00	17.84	0.1433	0.3172
SCAQS4	Anaheim, CA	11/11/87	12/11/87	0000-2400	6	83.5	1.136	30.56	4.28	9.93	5.45	13.88	0.1711	0.4302
SCAQS4	Rubidoux, CA	11/11/87	12/11/87	0000-2400	6	85.8	0.81	29.04	3.13	7.51	5.54	13.61	0.5893	1.3430

Table 2-3 (continued)
Average PM$_{2.5}$ Composition (μg/m^3) in Selected Urban and Non-Urban U.S. Areas

Study	Site	V	Cr	Mn	Fe	Ni	Cu	Zn	Se	Br	Rb	Sr	Zr	Ba	Pb
AUSPEX1	Point Reyes, CA	0.0010	0.0000	0.0010	0.0180	0.0000	0.0020	#N/A	0.0000	0.0010	0.0000	0.0010	0.0000	0.0430	0.0010
AUSPEX1	Altamont Pass, CA	0.0010	0.0010	0.0030	0.1110	0.0010	0.0030	#N/A	0.0020	0.0030	0.0010	0.0010	0.0000	0.0520	0.0110
AUSPEX1	Pacheco Pass, CA	0.0010	0.0010	0.0020	0.1060	0.0010	0.0020	#N/A	0.0000	0.0030	0.0000	0.0010	0.0010	0.0560	0.0030
AUSPEX1	Crows Landing, CA	0.0030	0.0030	0.0210	1.1840	0.0030	0.0040	#N/A	0.0020	0.0060	0.0020	0.0050	0.0020	0.0620	0.0100
AUSPEX1	Academy, CA	0.0010	0.0000	0.0090	0.3910	0.0010	0.0090	#N/A	0.0000	0.0060	0.0010	0.0020	0.0010	0.0470	0.0050
AUSPEX1	Buttonwillow, CA	0.0030	0.0000	0.0070	0.4270	0.0040	0.0050	#N/A	0.0010	0.0070	0.0010	0.0030	0.0010	0.0660	0.0070
AUSPEX1	Edison, CA	0.0030	0.0000	0.0330	1.9530	0.0030	0.0100	#N/A	0.0010	0.0100	0.0040	0.0110	0.0030	0.0700	0.0090
AUSPEX1	Caliente, CA	0.0020	0.0000	0.0090	0.5410	0.0010	0.0040	#N/A	0.0000	0.0070	0.0010	0.0030	0.0010	0.0570	0.0070
AUSPEX1	Sequoia, CA	0.0010	0.0000	0.0010	0.0740	0.0000	0.0030	#N/A	0.0000	0.0040	0.0010	0.0010	0.0010	0.0480	0.0040
AUSPEX1	Yosemite, CA	0.0010	0.0000	0.0030	0.1600	0.0000	0.0010	#N/A	0.0000	0.0030	0.0010	0.0010	0.0010	0.0520	0.0030
IMS95	Bakersfield, CA	0.0010	0.0003	0.0023	0.1231	0.0011	0.0115	0.0391	0.0009	0.0111	0.0004	0.0007	0.0004	0.0111	0.0134
IMS95	Fresno, CA	0.0005	0.0005	0.0015	0.0850	0.0004	0.0055	0.0392	0.0009	0.0095	0.0005	0.0004	0.0008	0.0105	0.0149
IMS95	Kern Wildlife Refuge, CA	0.0007	0.0003	0.0010	0.0517	0.0014	0.0044	0.0284	0.0011	0.0036	0.0002	0.0004	0.0002	0.0071	0.0053
IMS95	Chowchilla, CA	0.0002	0.0001	0.0005	0.0232	0.0008	0.0037	0.0175	0.0010	0.0044	0.0001	0.0003	0.0002	0.0070	0.0071
SCAQS4	Burbank, CA	0.0059	0.0191	0.0129	0.0718	0.0042	0.0183	0.0178	0.0121	0.0155	#N/A	0.0210	#N/A	0.0145	0.0475
SCAQS4	Downtown Los Angeles, CA	0.0055	0.0216	0.0156	0.0987	0.0049	0.0629	0.0896	0.0125	0.0133	#N/A	0.0189	#N/A	0.0149	0.0381
SCAQS4	Hawthorne, CA	0.0061	0.0198	0.0130	0.0287	0.0057	0.0727	0.0585	0.0133	0.0103	#N/A	0.0207	#N/A	0.0061	0.0334
SCAQS4	Long Beach, CA	0.0068	0.0220	0.0143	0.0714	0.0110	0.0047	0.0267	0.0102	0.0105	#N/A	0.0206	#N/A	0.0076	0.0356
SCAQS4	Anaheim, CA	0.0056	0.0185	0.0122	0.0296	0.0045	0.0396	0.0333	0.0115	0.0113	#N/A	0.0176	#N/A	0.0075	0.0346
SCAQS4	Rubidoux, CA	0.0054	0.0184	0.0147	0.2062	0.0047	0.0165	0.0131	0.0123	0.0121	#N/A	0.0199	#N/A	0.0088	0.0350
SCAQS4	San Nicolas Island, CA	0.0049	0.0215	0.0124	0.0249	0.0047	0.0629	0.0395	0.0103	0.0102	#N/A	0.0200	#N/A	0.0040	0.0319
SCAQS4	Azusa, CA	0.0057	0.0180	0.0157	0.2819	0.0045	0.0134	0.0553	0.0115	0.0148	#N/A	0.0192	#N/A	0.0265	0.0486
SCAQS4	Claremont, CA	0.0053	0.0261	0.0141	0.1123	0.0078	0.0725	0.0778	0.0155	0.0107	#N/A	0.0193	#N/A	0.0128	0.0341
SCAQS4	Burbank, CA	0.0059	0.0335	0.0359	0.3525	0.0051	0.1797	0.2055	0.0134	0.0791	#N/A	0.0357	#N/A	0.0361	0.2308
SCAQS4	Downtown Los Angeles, CA	0.0066	0.0247	0.0427	0.5566	0.0070	0.2728	0.2982	0.0105	0.0652	#N/A	0.0280	#N/A	0.0430	0.1853
SCAQS4	Hawthorne, CA	0.0132	0.0313	0.0368	0.3745	0.0118	0.5184	0.4525	0.0116	0.0553	#N/A	0.0207	#N/A	0.0302	0.1584
SCAQS4	Long Beach, CA	0.0089	0.0211	0.0415	0.3297	0.0047	0.0741	0.1921	0.0127	0.0843	#N/A	0.0252	#N/A	0.0276	0.2134
SCAQS4	Anaheim, CA	0.0079	0.0363	0.0335	0.3596	0.0048	0.0817	0.1368	0.0118	0.0673	#N/A	0.0334	#N/A	0.0212	0.1878
SCAQS4	Rubidoux, CA	0.0056	0.0328	0.0447	0.8992	0.0045	0.2080	0.1932	0.0113	0.0662	#N/A	0.0224	#N/A	0.0300	0.1451

Table 2-3 (continued)
Average PM$_{2.5}$ Composition (μg/m^3) in Selected Urban and Non-Urban U.S. Areas

Study	Site	Sampling StartDate	Sampling EndDate	Integration Time (h)	# in Avg	Mass	Cl-	NO3-	SO4=	NH4+	EC	OC	Al	Si
Phoenix PM10	Estrella Park, AZ	09/25/89	01/21/90	0000-2400	10	18.48	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	0.3425	0.6966
Phoenix PM10	Gunnery Range, AZ	09/25/89	01/21/90	0000-2400	10	7.59	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	0.1380	0.3896
Phoenix PM10	Pinnacle Peak, AZ	09/25/89	01/21/90	0000-2400	11	4.63	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	0.0382	0.1110
Phoenix Urban Haze	ICA, CA	09/25/89	01/22/90	0600-1200	43	27.87	0.0990	3.9750	1.6450	1.2060	8.0810	8.65	0.2120	0.7090
Phoenix Urban Haze	Valley Bank, AZ	09/25/89	01/22/90	0600-1200	43	15.09	0.0560	2.9800	1.1330	0.8720	3.6760	5.58	0.1420	0.4320
Phoenix Urban Haze	West Phoenix, AZ	09/25/89	01/22/90	0600-1200	44	30.04	0.0980	4.3010	1.4970	1.1350	8.8370	9.92	0.1860	0.6190
Phoenix Urban Haze	South Scottsdale, AZ	09/25/89	01/22/90	0600-1200	41	16.32	0.0570	2.1420	1.0130	0.6780	4.3360	5.58	0.1620	0.5320
Phoenix Urban Haze	GM Proving Grounds, AZ	09/25/89	01/22/90	0600-1200	46	10.16	0.0020	0.2020	1.3000	0.5150	2.1500	0.84	0.3150	0.7810
Phoenix Urban Haze	ICA, AZ	09/25/89	01/22/90	1200-1800	43	15.35	0.0170	4.4380	1.2000	0.9590	3.0580	6.16	0.1700	0.5350
Phoenix Urban Haze	Valley Bank, AZ	09/25/89	01/22/90	1200-1800	44	14.67	0.0490	4.2180	1.0870	0.9330	2.2930	4.99	0.1830	0.4850
Phoenix Urban Haze	West Phoenix, AZ	09/25/89	01/22/90	1200-1800	44	16.52	0.0240	4.5720	1.1230	0.8630	2.7630	6.13	0.1860	0.5650
Phoenix Urban Haze	South Scottsdale, AZ	09/25/89	01/22/90	1200-1800	40	15.37	0.0570	3.6820	1.6990	0.8820	2.9840	5.51	0.1760	0.5300
Pilot Tucson PM	Downtown Tucson, AZ	09/29/89	01/23/90	0000-2400	7	14.37	0.0520	0.5100	1.4100	0.5800	2.1000	4.26	0.1300	0.3900
Pilot Tucson PM	22nd and Craycroft, AZ	09/29/89	01/23/90	0000-2400	6	8.44	0.0430	0.6900	1.2300	0.6400	1.7400	2.54	0.0850	0.2400
Pilot Tucson PM	Corona de Tucson, AZ	09/29/89	01/23/90	0000-2400	6	3.52	N/Ab	N/A	N/A	N/A	N/A	N/A	0.0380	0.1600
Denver Brown Cloud	Auraria, CO	11/02/87	01/31/88	0900-0900	133	20.39	#N/A	3.5490	1.5510	1.6260	4.8640	7.95	0.0350	0.2700
Denver Brown Cloud	Federal, CO	11/02/87	01/31/88	0900-0900	138	16.42	#N/A	3.2290	1.3840	1.4760	3.4000	6.37	0.0320	0.2640
Denver Brown Cloud	Welby, CO	11/02/87	01/31/88	0900-0900	135	22.20	#N/A	5.0900	1.7070	2.1320	4.9620	7.42	0.0430	0.2820
Mt. Zirkel	Buffalo Pass, CO	02/16/95	10/29/95	0600-1800	64	4.70	0.0094	0.0882	0.8065	0.2673	0.2572	0.92	0.0750	0.1737
Mt. Zirkel	Gilpin Creek, CO	02/16/95	10/29/95	0600-1800	24	4.32	0.0491	0.2189	0.9017	0.2587	0.9628	1.20	0.0312	0.1120
Mt. Zirkel	Juniper Mountain, CO	02/16/95	10/29/95	0600-1800	31	4.50	0.0057	0.0651	0.8804	0.2938	0.4232	1.27	0.1273	0.1347
Mt. Zirkel	Baggs, CO	02/16/95	10/29/95	0600-1800	43	3.83	0.0123	0.0795	0.8641	0.2372	0.4106	2.10	0.0516	0.1308
Mt. Zirkel	Hayden VOR, CO	02/16/95	10/29/95	0600-1800	61	5.67	0.0078	0.1030	1.0863	0.4948	0.4447	1.85	0.1150	0.2532
Mt. Zirkel	Hayden Waste Water, CO	02/16/95	10/29/95	0600-1800	43	5.40	0.0179	0.2458	1.0763	0.3512	0.4789	1.69	0.1015	0.1583
Robbins	Alsip, IL	10/12/95	09/30/96	0000-2400	29	17.47	NA	NA	NA	#N/A	NA	NA	0.0329	0.1045
Robbins	Breman, IL	10/12/95	09/30/96	0000-2400	28	18.87	NA	NA	NA	#N/A	NA	NA	0.0361	0.1242
Robbins	Meadow Lane, IL	10/12/95	09/30/96	0000-2400	27	16.97	NA	NA	NA	#N/A	NA	NA	0.0572	0.1280
Robbins	Eisenhower, IL	10/12/95	09/30/96	0000-2400	28	18.08	NA	NA	NA	#N/A	NA	NA	0.0613	0.1473

Table 2-3 (continued)
Average $PM_{2.5}$ Composition ($\mu g/m^3$) in Selected Urban and Non-Urban U.S. Areas

Study	Site	V	Cr	Mn	Fe	Ni	Cu	Zn	Se	Br	Rb	Sr	Zr	Ba	Pb
Phoenix PM10	Estrella Park, AZ	0.0013	0.0016	0.0086	0.1879	0.0012	0.0156	0.1465	0.0012	0.0059	0.0004	0.0023	0.0005	0.0112	0.0247
Phoenix PM10	Gunnery Range, AZ	0.0011	0.0007	0.0044	0.0993	0.0008	0.0026	0.0083	0.0004	0.0031	0.0001	0.0012	0.0003	0.0081	0.0104
Phoenix PM10	Pinnacle Peak, AZ	0.0005	0.0005	0.0021	0.0349	0.0002	0.0029	0.0044	0.0005	0.0022	0.0001	0.0004	0.0001	0.0072	0.0061
Phoenix Urban Haze	ICA, CA	0.0050	0.0070	0.0330	0.3440	0.0080	0.0200	0.1090	0.0020	0.0170	0.0010	0.0050	0.0020	0.0230	0.0550
Phoenix Urban Haze	Valley Bank, AZ	0.0050	0.0040	0.0170	0.1940	0.0050	0.0160	0.0570	0.0030	0.0080	0.0010	0.0030	0.0010	0.0220	0.0290
Phoenix Urban Haze	West Phoenix, AZ	0.0060	0.0060	0.0470	0.3210	0.0060	0.0160	0.1860	0.0020	0.0150	0.0010	0.0060	0.0010	0.0230	0.0540
Phoenix Urban Haze	South Scottsdale, AZ	0.0050	0.0020	0.0130	0.1960	0.0020	0.0110	0.0300	0.0020	0.0080	0.0010	0.0020	0.0010	0.0240	0.0280
Phoenix Urban Haze	GM Proving Grounds, AZ	0.0030	0.0020	0.0080	0.1710	0.0010	0.0140	0.0180	0.0040	0.0030	0.0010	0.0020	0.0010	0.0350	0.0200
Phoenix Urban Haze	ICA, AZ	0.0040	0.0030	0.0160	0.2160	0.0030	0.0130	0.0450	0.0020	0.0060	0.0010	0.0030	0.0010	0.0310	0.0280
Phoenix Urban Haze	Valley Bank, AZ	0.0050	0.0030	0.0130	0.1920	0.0030	0.0130	0.0440	0.0010	0.0050	0.0010	0.0020	0.0010	0.0210	0.0200
Phoenix Urban Haze	West Phoenix, AZ	0.0040	0.0030	0.0170	0.2190	0.0030	0.0090	0.0450	0.0020	0.0060	0.0010	0.0020	0.0010	0.0300	0.0260
Phoenix Urban Haze	South Scottsdale, AZ	0.0050	0.0030	0.0150	0.2270	0.0020	0.0110	0.0350	0.0010	0.0060	0.0010	0.0020	0.0010	0.0310	0.0280
Pilot Tucson PM	Downtown Tucson, AZ	0.0028	0.0020	0.0140	0.1200	0.0008	0.0110	0.0190	0.0012	0.0076	0.0004	0.0061	).0006a	<0.016a	0.0290
Pilot Tucson PM	22nd and Craycroft, AZ	0.0012	0.0007	0.0078	0.0700	0.0004	0.0075	0.0110	0.0010	0.0040	0.0003	0.0009	).0006a	<0.016a	0.0180
Pilot Tucson PM	Corona de Tucson, AZ	0.0006	).0004a	0.0024	0.0420	0.0003	0.0033	0.0058	).0004a	0.0016	).0002a	).0004a	).0006a	<0.016a	0.0051
Denver Brown Cloud	Auraria, CO	#N/A	0.0060	0.0150	0.1320	0.0040	0.0220	0.0410	0.0010	0.0220	#N/A	0.0010	#N/A	#N/A	0.0880
Denver Brown Cloud	Federal, CO	#N/A	0.0020	0.0090	0.0860	0.0010	0.0210	0.0260	0.0010	0.0110	#N/A	0.0010	#N/A	#N/A	0.0550
Denver Brown Cloud	Welby, CO	#N/A	0.0020	0.0120	0.1160	0.0010	0.0070	0.0270	0.0010	0.0220	#N/A	0.0010	#N/A	#N/A	0.0820
Mt. Zirkel	Buffalo Pass, CO	0.0003	0.0003	0.0012	0.0614	0.0002	0.0026	0.0064	0.0002	0.0015	0.0002	0.0005	0.0002	0.0069	0.0009
Mt. Zirkel	Gilpin Creek, CO	0.0005	0.0009	0.0009	0.0304	0.0003	0.0006	0.0017	0.0002	0.0013	0.0001	0.0005	0.0011	0.0269	0.0012
Mt. Zirkel	Juniper Mountain, CO	0.0002	0.0005	0.0008	0.0360	0.0002	0.0020	0.0248	0.0001	0.0018	0.0001	0.0005	0.0002	0.0059	0.0010
Mt. Zirkel	Baggs, CO	0.0006	0.0002	0.0007	0.0320	0.0001	0.0005	0.0050	0.0004	0.0016	0.0001	0.0005	0.0003	0.0050	0.0007
Mt. Zirkel	Hayden VOR, CO	0.0007	0.0004	0.0015	0.0710	0.0003	0.0033	0.0119	0.0008	0.0019	0.0002	0.0012	0.0004	0.0048	0.0013
Mt. Zirkel	Hayden Waste Water, CO	0.0006	0.0003	0.0010	0.0545	0.0005	0.0027	0.0101	0.0008	0.0085	0.0001	0.0010	0.0004	0.0056	0.0015
Robbins	Alsip, IL	0.0010	0.0013	0.0065	0.1337	0.0009	0.0141	0.0499	0.0015	0.0044	0.0004	0.0006	0.0001	0.0066	0.0176
Robbins	Breman, IL	0.0012	0.0011	0.0100	0.1898	0.0008	0.0059	0.0741	0.0019	0.0049	0.0005	0.0007	0.0002	0.0106	0.0233
Robbins	Meadow Lane, IL	0.0012	0.0010	0.0074	0.1672	0.0009	0.0058	0.0554	0.0016	0.0044	0.0005	0.0007	0.0025	0.0073	0.0194
Robbins	Eisenhower, IL	0.0015	0.0012	0.0085	0.1756	0.0012	0.0067	0.0547	0.0018	0.0045	0.0005	0.0006	0.0001	0.0123	0.0194

Table 2-3 (continued)
Average $PM_{2.5}$ Composition ($\mu g/m^3$) in Selected Urban and Non-Urban U.S. Areas

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[a] Chow et al. (1996a).

[b] Chow and Egami (1997).

[c] Chow et al. (1994a).

[d] Chow et al. (1991).

[e] Watson et al. (1990, 1991a).

[f] Chow et al. (1992c).

[g] Watson et al. (1988a, 1988b, 1988c).

[h] Watson et al. (1996a).

[i] Watson et al. (1997d).

Table 2-4
Receptor Model Source Contributions to PM$_{10}$

Sampling Site	Time Period	Primary Geological	Primary Construction	Primary Motor Vehicle Exhaust	Primary Vegetative Burning	Secondary Ammonium Sulfate	Secondary Ammonium Nitrate	Misc. Source 1	Misc. Source 2	Misc. Source 3	Misc. Source 4	Measured PM$_{10}$ Concentration
Central Phoenix, AZ (Chow et al., 1991)	Winter 1989-90	33.0	0.0	25.0	2.3	0.2	2.8	0.0	0.0	0.0	0.0	64.0
Corona de Tucson, AZ (Chow et al., 1992c)	Winter 1989-90	17.0	0.0	1.6	0.0	1.9	0.0	0.0	0.0	0.0	0.0	19.1
Craycroft, AZ (Chow et al., 1992c)	Winter 1989-90	13.0	0.0	8.3	0.0	0.7	0.6	1.2[a]	0.0	0.0	0.0	23.4
Downtown Tucson, AZ (Chow et al., 1992c)	Winter 1989-90	26.0	5.1	14.0	0.0	1.0	0.2	1.3[a]	0.0	0.0	0.0	48.0
Hayden 1, AZ (Garfield) (Ryan et al., 1988)	1986	5.0	2.0[b]	0.0	0.0	4.0	0.0	74.0[c]	5.0[d]	1.0[e]	0.0	105.0
Hayden 2, AZ (Jail) (Ryan et al., 1988)	1986	21.0	4.0[b]	0.0	0.0	4.0	0.0	28.0[c]	0.0	1.0[e]	0.0	59.0
Orange Grove, AZ (Chow et al., 1992a)	Winter 1989-90	20.0	0.0	15.0	0.0	0.4	0.4	0.0	0.0	0.0	0.0	34.2
Phoenix, AZ (Estrella Park) (Chow et al., 1991)	Winter 1989-90	37.0	0.0	10.0	0.9	1.6	0.0	0.0	0.0	0.0	0.0	55.0
Phoenix, AZ (Gunnery Rg.) (Chow et al., 1991)	Winter 1989-90	20.0	0.0	5.5	0.0	1.0	0.0	0.0	0.0	0.0	0.0	27.0
Phoenix, AX (Pinnacle Pk.) (Chow et al., 1991)	Winter 1989-90	7.0	0.0	2.9	1.0	0.9	0.0	0.0	0.0	0.0	0.0	12.0
Rillito, AZ (Thanukos et al., 1992)	1988	42.7	13.8[b]	1.2[f]	0.0	0.0	0.0	11.6[g]	0.0	0.0	0.0	79.5
Scottsdale, AZ (Chow et al., 1991)	Winter 1989-90	25.0	0.0	19.0	7.4	0.6	3.6	0.0	0.0	0.0	0.0	55.0
West Phoenix, AZ (Chow et al., 1991)	Winter 1989-90	30.0	0.0	25.0	10.0	0.4	3.1	0.0	0.0	0.0	0.0	69.0
Anacapa Island, CA (Chow et al., 1996b)		2.2	0.0	4.9	0.0	3.4	1.0	9.6[h]	0.0	0.0	0.0	26.0
Anaheim, CA (Gray et al., 1988)	1986	21.2	0.0	4.1[i]	0.0	7.0	9.8	0.4[j]	1.4[h]	8.2[k]	0.0	52.1
Anaheim, CA (Summer) (Watson et al., 1994a)	Summer 1987	11.4	0.0	8.5	0.0	9.0	2.9	0.0[j]	6.5[h]	0.0	0.0	51.3
Anaheim, CA (Fall) (Watson et al., 1994c)	Fall 1987	13.2	0.0	37.2	0.0	3.7	38.5	0.0[j]	3.1[h]	0.0	0.0	104.0
Azusa, CA (Summer) (Watson et al., 1994c)	Summer 1987	34.9	0.0	15.9	0.0	11.4	6.1	0.0[j]	5.7[h]	0.0	0.0	92.1
Bakersfield, CA (Magliano, 1988)	1986	27.4	3.0	5.5	9.6[l]	5.6	0.0	0.5[j]	0.0	0.0	0.0	67.6
Bakerfield, CA (Chow et al., 1992a)	1988-89	42.9	1.6	7.7	6.5	5.5	12.7	1.0[m]	1.5[n]	0.6[k]	0.0	79.6
Burbank, CA (Gray et al., 1988)	1986	21.3	0.0	6.1[i]	0.0	7.2	10.2	0.1[j]	0.9[h]	9.8[k]	0.0	56.6
Burbank, CA (Summer) (Watson et al., 1994c)	Summer 1987	14.0	0.0	17.0	0.0	12.4	6.5	0.0[j]	5.7[h]	0.0	0.0	72.3
Burbank, CA (Fall) (Watson et al., 1994c)	Fall 1987	11.0	0.0	39.1	0.0	3.1	25.1	0.0[j]	1.9[h]	0.0	0.0	94.8
Chula Vista 1, CA (Bayside) (Cooper et al., 1988)	1986	6.7	0.0	0.8	0.0	7.5	0.0	0.4[j]	2.7[h]	2.0[k]	0.0	28.8
Chula Vista 2, CA (Del Ray) (Cooper et al., 1988)	1986	8.2	0.3	1.5	0.0	8.9	0.0	0.6[j]	1.8[h]	0.0	0.0	31.1
Chula Vista 3, CA (Cooper et al., 1988)	1986	9.7	0.3	1.4	0.0	8.2	0.0	0.6[j]	1.7[h]	0.0	0.0	29.6
Claremont, CA (Summer) (Watson et al., 1994c)	Summer 1987	19.4	0.0	14.4	0.0	9.5	6.3	0.0[j]	4.7[h]	0.0	0.0	70.0
Crows Landing, CA (Chow et al., 1992a)	1988-89	32.2	0.0	2.2	3.4	2.8	6.5	0.5[m]	1.5[n]	1.2[k]	0.0	52.5
Downtown Los Angeles, CA (Gray et al., 1988)	1986	23.8	0.0	6.4[i]	0.0	7.6	11.2	0.0	1.3[h]	7.9[k]	0.0	60.2
Downtown Los Angeles, CA (Summer) (Watson et al., 1994c)	Summer 1987	12.7	0.0	16.2	0.0	13.0	4.4	0.0[j]	6.5[h]	0.0	0.0	67.6
Downtown Los Angeles, CA (Fall) (Watson et al., 1994c)	Fall 1987	9.4	0.0	41.1	0.0	3.9	27.5	0.0[j]	1.8[h]	0.0	0.0	98.6
Fellows, CA (Chow et al., 1992a)	1988-89	29.0	1.4	2.1	3.4	5.1	7.5	7.0[m]	1.4[n]	1.4[k]	0.0	54.6
Fresno, CA (Magliano, 1988)	1986	17.1	0.7	4.0	9.2[l]	1.8	0.0	0.1[j]	0.0	0.0	0.0	48.1
Fresno, CA (Chow et al., 1992a)	1988-89	31.8	0.0	6.8	5.1	3.6	10.4	0.3[m]	1.0[n]	0.1[k]	0.0	71.5
Hawthorne, CA (Summer) (Watson et al., 1994c)	Summer 1987	7.5	0.0	5.6	0.0	15.0	0.6	0.0[j]	7.0[h]	0.0	0.0	45.9
Hawthorne, CA (Fall) (Watson et al., 1994c)	Fall 1987	8.9	0.0	35.1	0.0	5.1	20.4	0.0[j]	3.7[h]	0.0	0.0	85.1
Indio, CA (Kim et al., 1992)		33.0	3.0	4.4	7.1	3.6	4.1	0.2[j]	1.0[h]	0.0	0.0	58.0

Table 2-4 (continued)
Receptor Model Source Contributions to PM_{10}

Sampling Site	Time Period	Primary Geological	Primary Construction	Primary Motor Vehicle Exhaust	Primary Vegetative Burning	Secondary Ammonium Sulfate	Secondary Ammonium Nitrate	Misc. Source 1	Misc. Source 2	Misc. Source 3	Misc. Source 4	Measured PM_{10} Concentration
Kern Wildlife Refuge, CA (Chow et al., 1992a)	1988-89	15.1	2.0	2.2	4.0	3.3	1.5	0.5[m]	1.5[n]	0.7[k]	0.0	47.8
Lennox, CA (Gray et al., 1988)	1986	16.0	0.1	4.6[i]	0.0	7.6	7.9	0.2[j]	3.1[h]	7.6[k]	0.0	46.9
Long Beach, CA (Gray et al., 1988)	1986	20.7	0.0	5.1[i]	0.0	8.0	9.2	0.1[j]	2.0[h]	6.4[k]	0.0	51.9
Long Beach, CA (Summer) (Watson et al., 1994c)	Summer 1987	11.1	0.0	6.3	0.0	10.9	0.8	0.1[j]	2.2[h]	0.0	0.0	46.1
Long Beach, CA (Fall) (Watson et al., 1994c)	Fall 1987	11.3	0.0	42.8	0.0	3.8	23.2	0.0[j]	2.7[h]	0.0	0.0	96.1
Magnolia, CA (Chow et al., 1992b)	1988	31.7	0.0	11.2	0.0	4.9	19.7	0.3[j]	1.2[h]	1.2[o]	0.0	66.0
Palm Springs, CA (Kim et al., 1992)		16.4	1.4	2.3	5.1	3.7	4.2	0.1[j]	0.5[h]	0.0	0.0	35.1
Riverside, CA (Chow et al., 1992b)	1988	32.6	0.0	7.0	0.0	4.8	21.4	0.3[j]	1.3[h]	1.1[o]	0.0	64.0
Rubidoux, CA (Gray et al., 1988)	1986	43.1	4.0[j]	5.6[i]	0.0	6.4	21.3	0.3[j]	1.0[h]	5.9[k]	0.0	87.4
Rubidoux, CA (Summer) (Watson et al., 1994b)	Summer 1987	34.9	4.5	17.3	0.0	9.5	27.4	0.0[j]	5.1[h]	0.0	0.0	114.8
Rubidoux, CA (Fall) (Watson et al., 1994b)	Fall 1987	19.2	16.1	30.3	0.0	2.1	31.6	0.0[j]	1.1[h]	0.0	0.0	112.0
Rubidoux, CA (Chow et al., 1992b)	1988	48.0	0.0	10.2	0.0	5.3	21.7	0.4[j]	1.5[h]	5.7[o]	0.0	87.0
San Jose, CA (4th St.) (Chow et al., 1995b)		13.1	0.0	9.2	31.3	2.3	13.3	0.9[h]	0.0	0.0	0.0	68.4
San Jose, CA (San Carlos St.) (Chow et al., 1995b)		11.8	0.0	8.9	31.3	2.1	12.8	0.7[h]	0.0	0.0	0.0	64.9
San Nicolas Island, CA (Summer) (Watson et al., 1994c)	Summer 1987	1.6	0.0	0.9	0.0	3.7	0.5	0.0[j]	4.3[h]	0.0	0.0	17.4
Santa Barbara, CA (Chow et al., 1996b)		9.5	0.0	14.7	0.0	3.2	1.0	6.4[h]	0.0	0.0	0.0	34.0
Santa Barbara, CA (GTC) (Chow et al., 1996b)		3.2	0.0	5.1	0.0	2.8	0.5	6.3[h]	0.0	0.0	0.0	20.5
Santa Maria, CA (Chow et al., 1996b)		7.4	0.0	7.6	0.0	3.1	1.4	5.7[h]	0.0	0.0	0.0	27.0
Santa Ynez, CA (Chow et al., 1996b)		4.6	0.0	6.8	0.0	2.2	0.6	4.0[h]	0.0	0.0	0.0	19.0
Stockton, CA (Chow et al., 1992a)	1989	34.4	0.5	5.2	4.8	3.1	7.0	0.7[m]	1.8[n]	0.0[k]	0.0	62.4
Upland, CA (Gray et al., 1988)	1986	25.4	0.4[j]	4.1[i]	0.0	6.4	14.5	0.6[j]	0.6[h]	7.8[k]	0.0	58.0
Vandenberg AFB, CA (Watt Road) (Chow et al., 1996b)		4.5	0.0	3.2	0.0	1.9	1.0	9.3[h]	0.0	0.0	0.0	20.6
Telluride 1, CO (Central) (Dresser and Baird, 1988)	Winter 1986	32.0	0.0	0.0	98.7	0.0	0.0	61.3[p]	0.0	0.0	0.0	208.0
Telluride 2, CO (Society Turn) (Dresser amd Baird, 1988)	Winter 1986	12.1	0.0	0.0	7.3	0.0	0.0	7.3[p]	0.0	0.0	0.0	27.0
Pocatello, ID (Houck et al., 1992)	1990	8.3	7.5[q]	0.1	0.0	0.0	0.0	0.0	0.0	84.1[r]	0.0	100.0
S. Chicago, IL (Hopke et al., 1988)	1986	27.2	2.4	2.8	0.0	15.4[s]	0.0	15.1[t]	2.2[u]	0.0	0.0	80.1
S.E. Chicago, IL (Vermette et al., 1992)	1988	14.7[v]	0.0	0.9[f]	0.0	7.7	0.0	0.8[t]	0.3[h]	1.1[w]	7.7[g]	41.0
Reno, NV (Non-sweeping) (Chow et al., 1990)	Winter 1987	9.7	0.0	8.7	0.1	0.6	0.2	0.0	0.0	0.0	0.0	20.4
Reno, NV (Sweeping) (Chow et al., 1990)	Winter 1987	11.8	0.0	11.0	1.2	0.8	0.2	0.0	0.0	0.0	0.0	24.9
Reno, NV (Chow et al., 1988)	1986-87	14.9	0.0	10.0	1.9	1.3	0.6	0.0	0.0	0.0	0.0	30.0
Sparks, NV (Chow et al., 1988)	1986-87	15.1	0.0	11.6	13.4	2.7	0.9	0.0	0.0	0.2[k]	0.0	41.0
Verdi, NV (Chow et al., 1988)	1986-87	7.8	0.0	4.0	1.1	0.9	0.1	0.0	0.0	0.0	0.0	15.0
Follansbee, OH (Skidmore et al., 1992)	1991	10.0	0.0	35.0	0.0	16.0	0.0	9.3[t]	0.0	0.0	0.0	66.0
Mingo, OH (Skidmore et al., 1992)	1991	12.0	0.0	14.0	4.1	15.0	0.0	3.4[t]	11.0[x]	0.0	0.0	60.0

Table 2-4 (continued)
Receptor Model Source Contributions to PM_{10}

Sampling Site	Time Period	Primary Geological	Primary Construction	Primary Motor Vehicle Exhaust	Primary Vegetative Burning	Secondary Ammonium Sulfate	Secondary Ammonium Nitrate	Misc. Source 1	Misc. Source 2	Misc. Source 3	Misc. Source 4	Measured PM_{10} Concentration
Sewage Plant, OH (Skidmore et al., 1992)	1991	22.0	0.0	12.0	0.0	13.0	0.0	6.6[t]	8.7[x]	0.0	0.0	62.0
Steubenville, OH (Skidmore et al., 1992)	1991	8.3	0.0	14.0	0.8	14.0	0.0	3.8[t]	5.0[x]	0.0	0.0	46.0
WTOV Tower, OH (Skidmore et al., 1992)	1991	7.4	0.0	16.0	0.2	15.0	0.0	3.4[t]	7.9[x]	0.0	0.0	49.0
Wuhan, China (Zelenka et al., 1992)		55.0	21.4	1.2	49.2[l]	28.1	17.0	49.5[y]	13.6[z]	1.2[aa]	0.0	224.9

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[a] Smelter background aerosol.
[b] Cement plant sources, including kiln stacks, gypsum pile, and kiln area.
[c] Copper ore.
[d] Copper tailings.
[e] Copper smelter building.
[f] Heavy-duty diesel exhaust emission.
[g] Background aerosol.
[h] Marine aerosol, road salt, and sea salt plus sodium nitrate.
[i] Motor vehicle exhaust from diesel and leaded gasoline.
[j] Residual oil combustion.
[k] Secondary organic carbon.
[l] Biomass burning.
[m] Primary crude oil.
[n] $NaCl + NaNO_3$.
[o] Lime.
[p] Road sanding material.
[q] Asphalt industry.
[r] Phosphorus/phosphate industry.
[s] Regional sulfate.
[t] Steel mills.
[u] Refuse incinerator.
[v] Local road dust, coal yard road dust, steel haul road dust.
[w] Incineration.
[x] Unexplained mass.
[y] Residential coal burning.
[z] Aluminum processing.
[aa] Primary lead smelter.

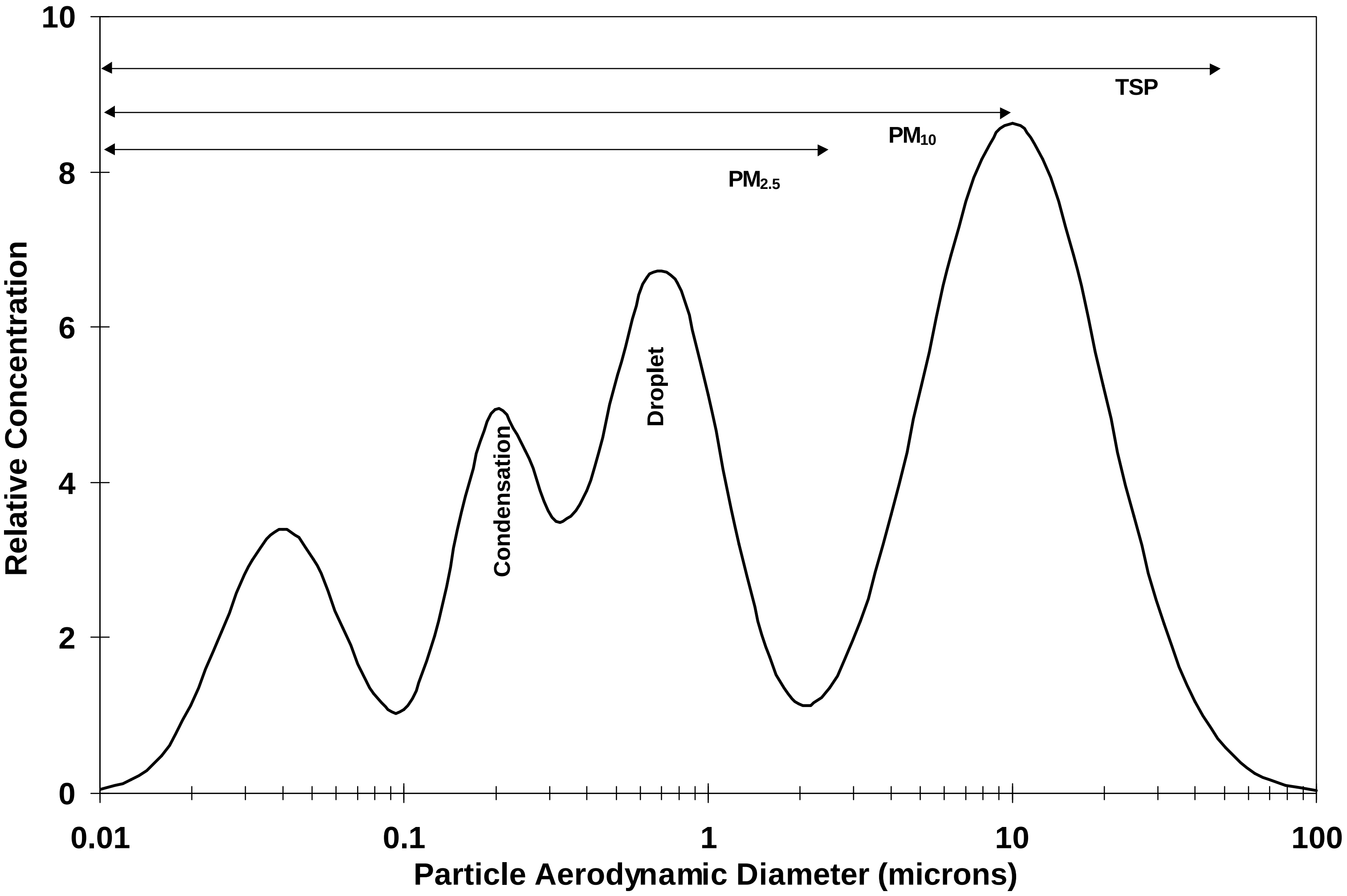


Figure 2-1. Idealized size distribution of particles in ambient air. The TSP and PM_{10} size fractions have been monitored to determine compliance with National Ambient Air Quality Standards. The $PM_{2.5}$ size fraction is commonly measured in source apportionment and visibility studies and is a potential size fraction to be measured for a new particle standard.

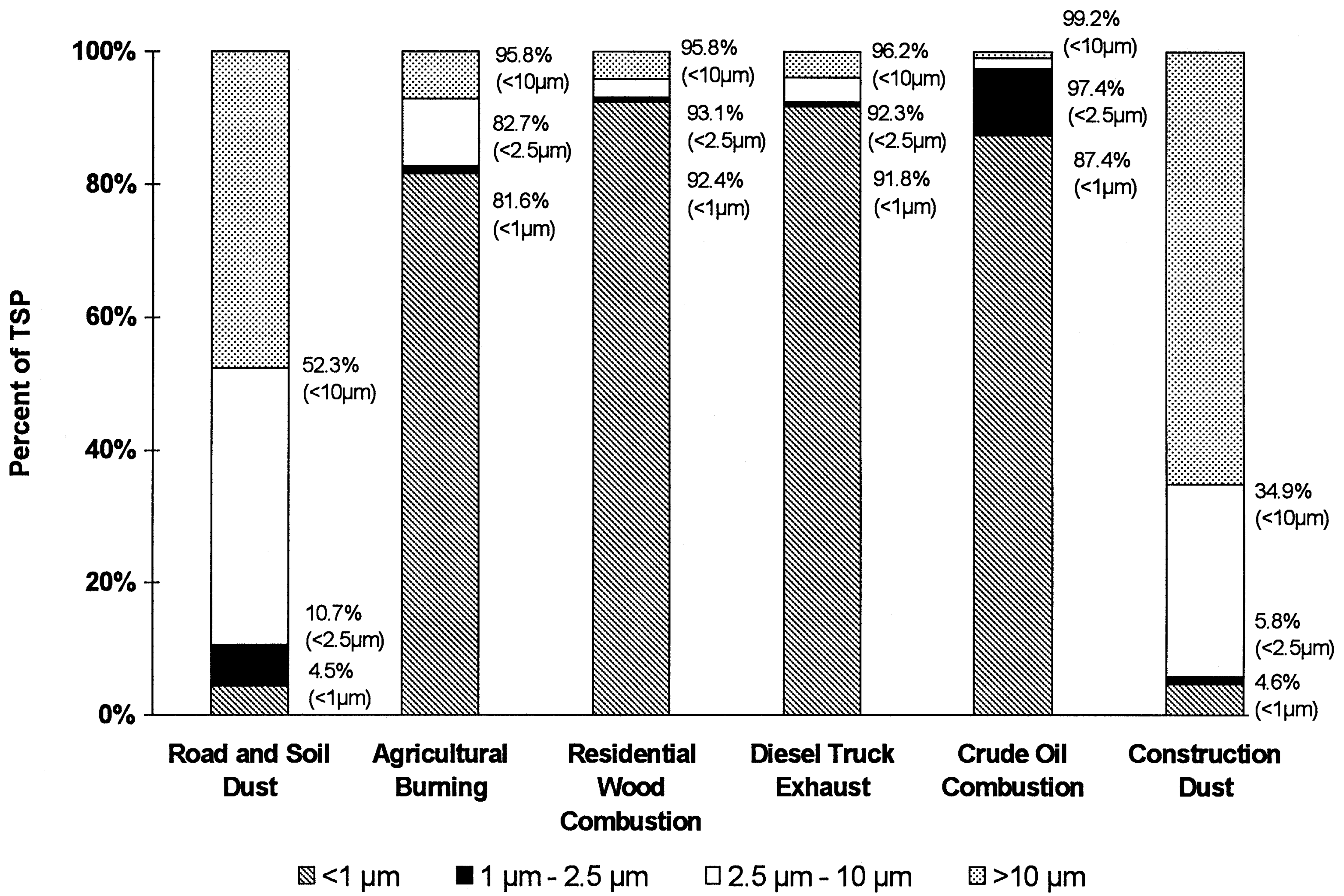


Figure 2-2. Size distributions of several particulate source emissions (Ahuja et al. 1989; Houck et al., 1989, 1990).

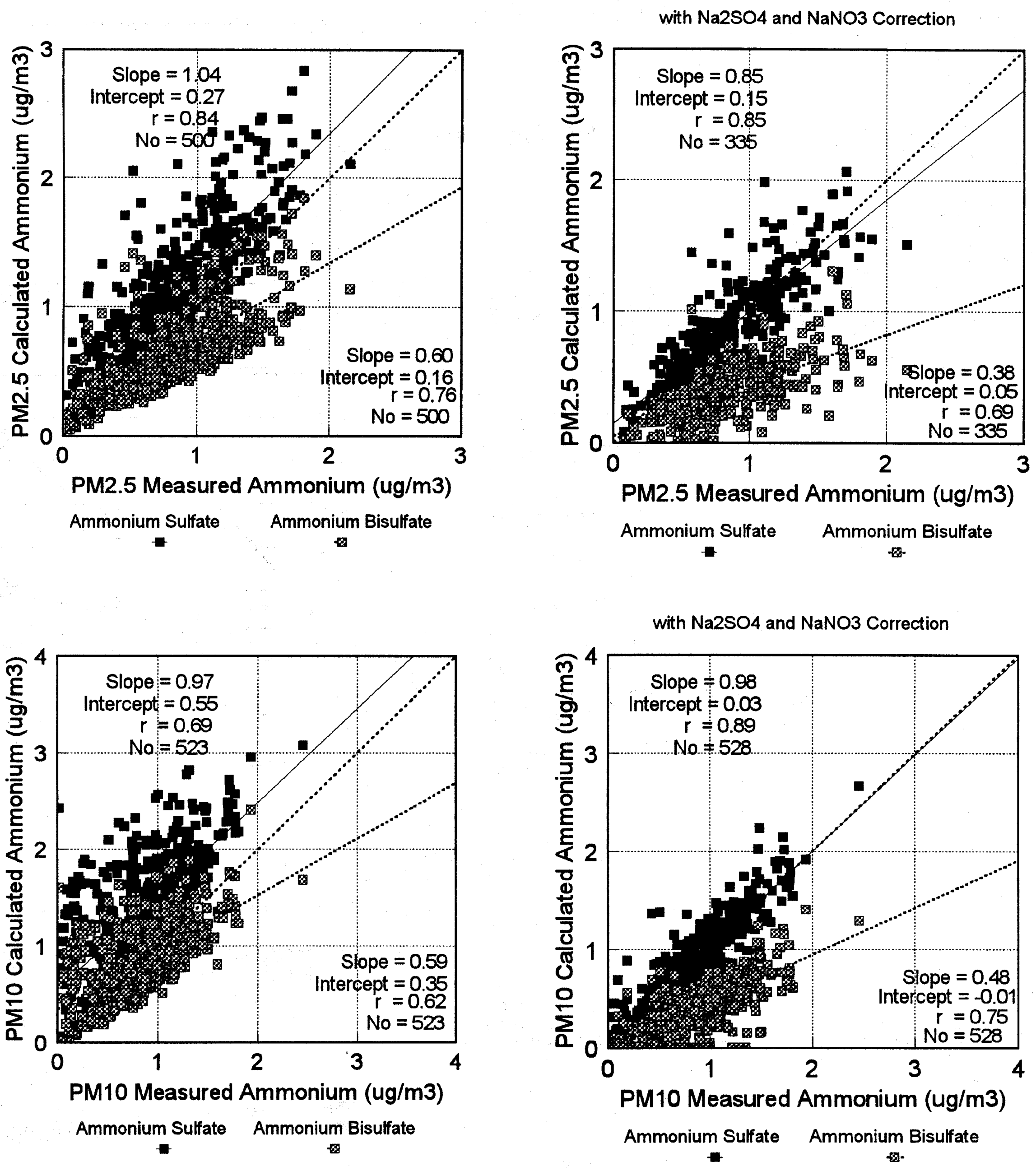
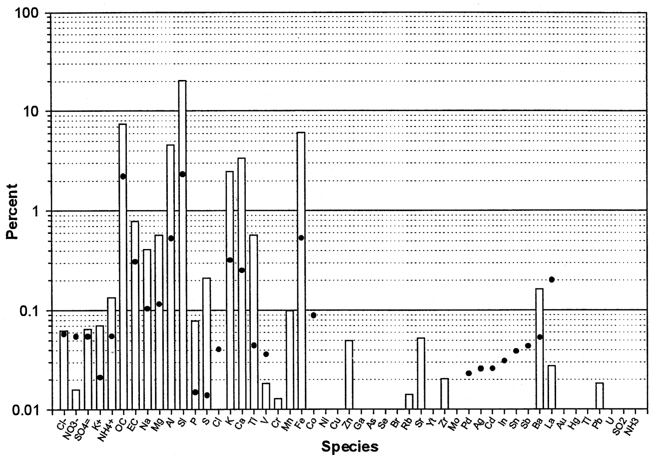
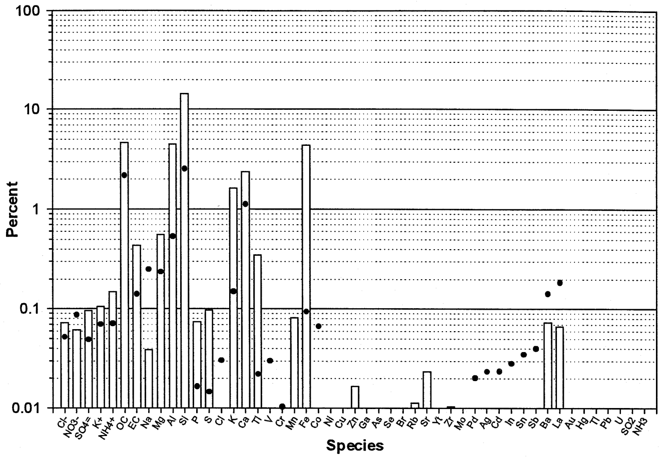


Figure 2-3. Comparison between calculated and measured ammonium at the ten SJVAQS/AUSPEX sites for $PM_{2.5}$ and PM_{10} size fractions.

(a) Paved Road Dust



(b) Unpaved Road Dust



(c) Natural Soil

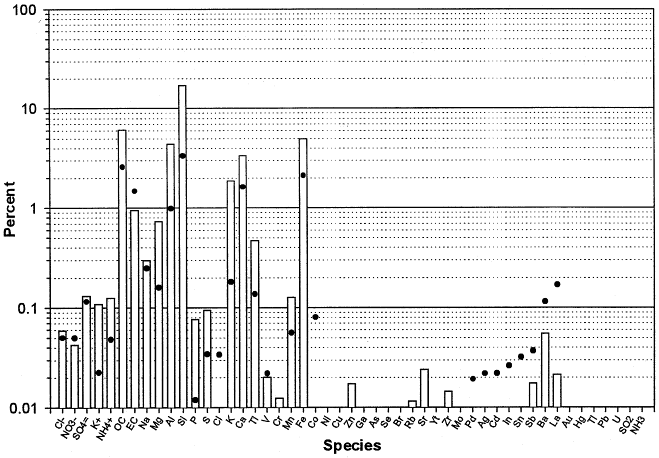
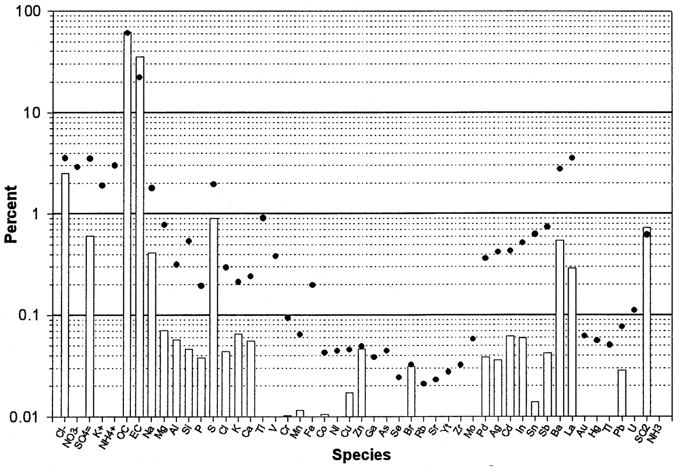
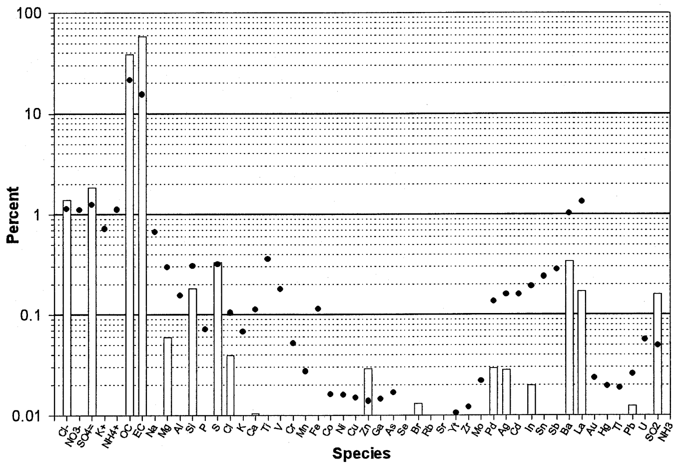


Figure 2-4. Geological material source profiles derived for northwestern Colorado.

(a) Local Traffic Emissions



(b) Highway Vehicle Emissions



(c) Composite Motor Vehicle Emissions

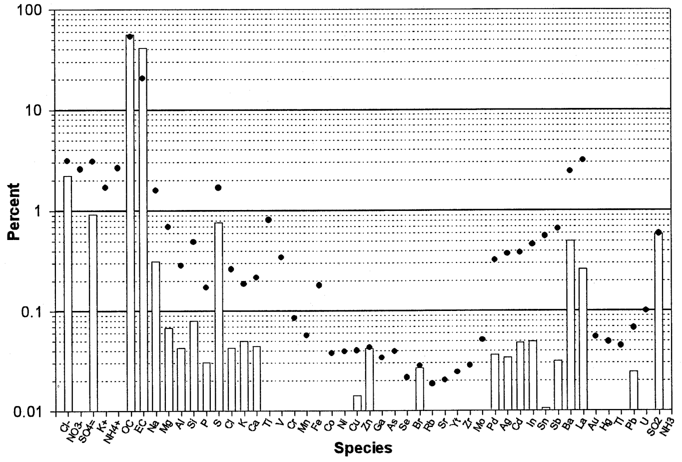
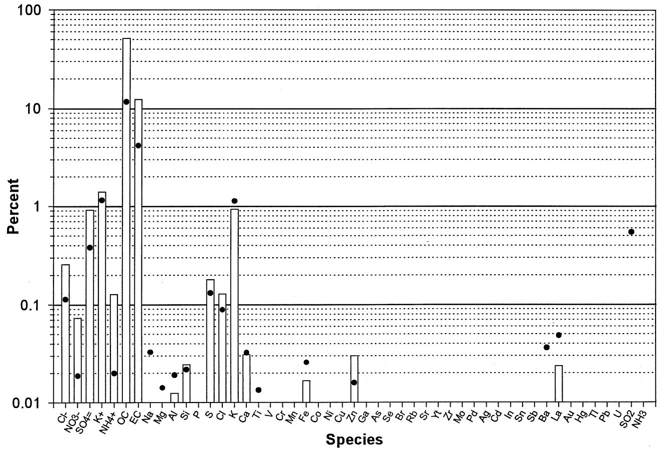
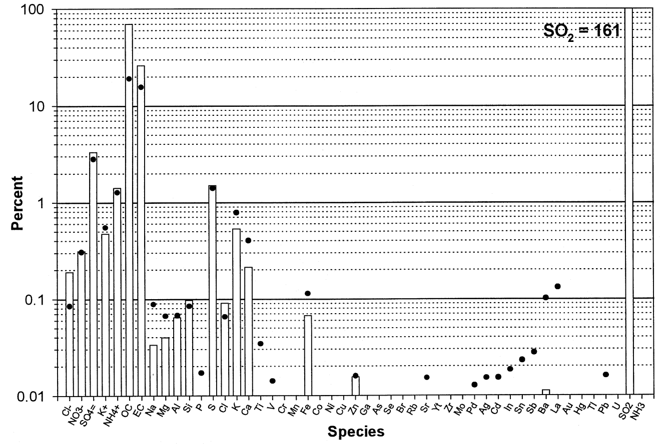


Figure 2-5. Motor vehicle emission source profiles derived for northwestern Colorado.

(a) Residential Wood Combustion



(b) Residential Coal Combustion



(c) Forest Fire Emissions

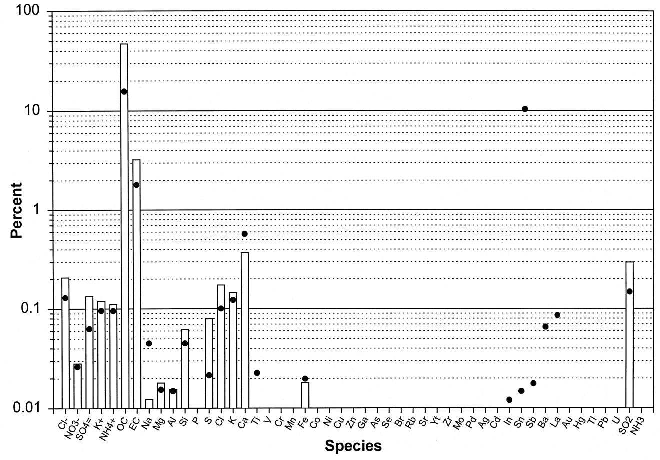
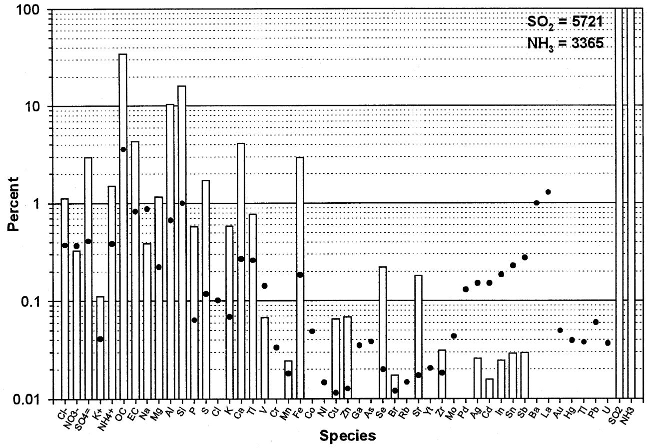
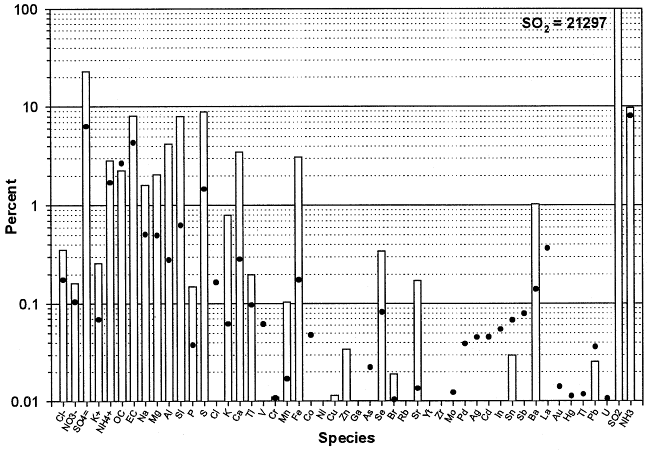


Figure 2-6. Emissions from burning source profiles derived for northwestern Colorado.

(a) Unit 1 Boiler



(b) Unit 2 Boiler



(c) Unit 3 Boiler

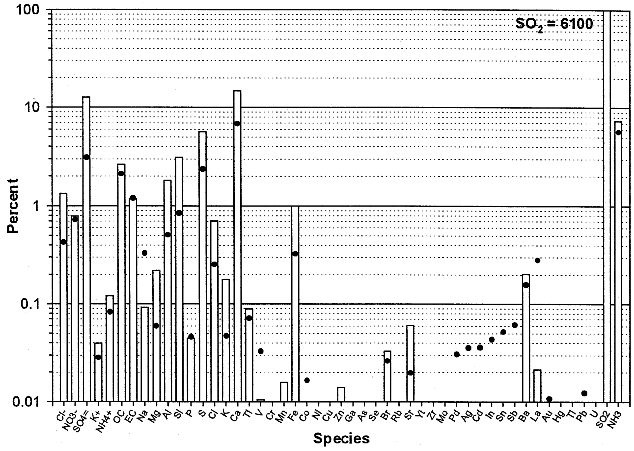


Figure 2-7. Coal-fired boiler source profiles derived for northwestern Colorado.

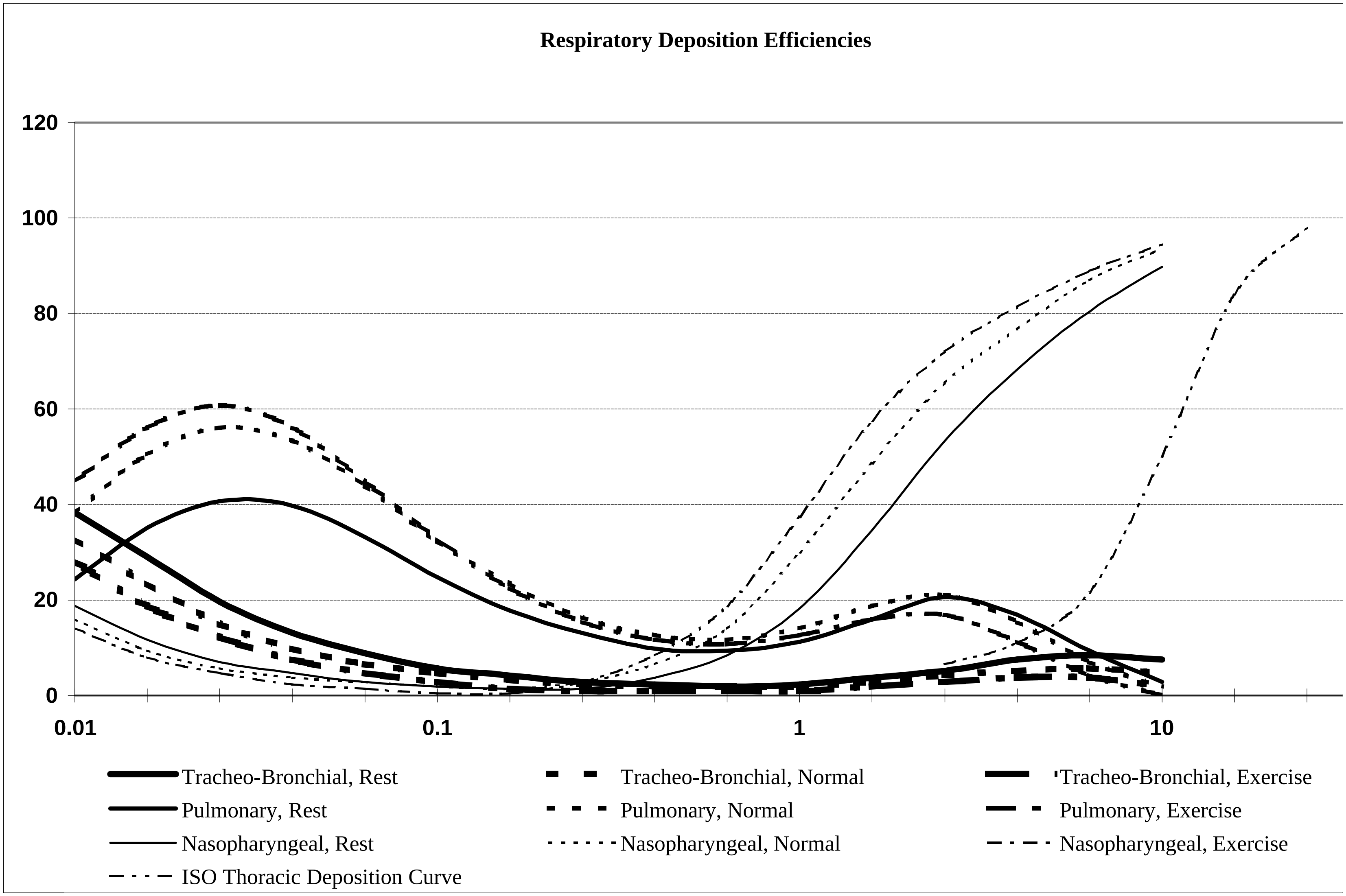


Figure 2-8. Deposition of particles inhaled into the human body.

3.0 PARTICLE SAMPLERS

Particle filtration samplers consist of combinations of size-selective inlets, filter media, filter holders, and flow movers/controllers. Denuder systems and absorbing materials that capture gases associated with volatile species such as ammonium nitrate and some organic compounds can be installed behind the size-selective inlet and behind the particle collection filter. The dimensions, materials, and construction of these components affect the particles that are measured.

This section summarizes the current knowledge of sampler components, describes compliance monitoring samplers (e.g., Federal Reference Method [FRM], Federal Equivalent Method [FEM], and Interagency Monitoring of Protected Visual Environments [IMPROVE]), and introduces monitors that have been used or might be applied for $PM_{2.5}$ chemical speciation.

3.1 Sampler Components

Tables 3-1 to 3-4 describe components available to construct particle samplers for both compliance and research monitoring. The following subsections summarize the measurement methods, operating principles, chemical/physical characteristics, and applications of these sampler components.

3.1.1 Size-Selective Inlets

Several size-selective inlets commonly used for aerosol sampling are summarized in Table 3-1. Hering (1995) lists other available inlets with various particle sizing characteristics that are often used in research studies and industrial hygiene applications. Sampling inlets are characterized by sampling effectiveness curves, such as that shown in Figure 3-1 for the WINS (Well Impactor-Ninety Six) $PM_{2.5}$ impactor specified for $PM_{2.5}$ FRMs. These curves are measured by presenting known concentrations of particles with selected aerodynamic diameters to the inlet at different wind velocities in a wind tunnel (U.S. EPA, 1987). While the effectiveness of PM_{10} inlets can be very sensitive to changes in wind speed for particles with aerodynamic diameters near 10 μm, the small inertia of particles with diameters of 2.5 μm results in more consistent penetration properties of $PM_{2.5}$ inlets under large range of wind speed and direction.

The aerodynamic diameter at which 50% of the sampled particles penetrate an inlet is termed the 50% cut-point (d_{50}). The ratio of the particle diameter for which 16% of sampled particles penetrate the inlet to the diameter at which 84% penetrate the inlet (d_{16}/d_{84}) is termed the slope. A smaller slope indicates a sharper cut-point, resulting in a better distinction between particles that are larger and smaller than d_{50}. A slope of unity indicates that 100% of the particles with aerodynamic diameter less than the cut-point pass through the inlet to the filter, and 0% of those larger than the cut-point are collected. No aerodynamic inlet can have such a sharp cut-point, and slopes typically range from 1.3 to 2, as shown in Table 3-1.

Figure 3-2 shows how an ambient size distribution is modified by passing through $PM_{2.5}$ and PM_{10} inlets. The PM_{10} inlet attenuates the collection of the coarse particle fraction, while the $PM_{2.5}$ practically eliminates it. Nevertheless, a small quantity of coarse particles are collected by a $PM_{2.5}$ sampler. The $PM_{2.5}$ sampling effectiveness curves in Figure 3-1 are similar to, in fact sharper than, the nasal deposition curves shown in Figure 2-8. $PM_{2.5}$ inlets with slopes exceeding unity probably simulate the different fractions of particles that penetrate into the human body to areas where they might cause harm.

Inlet flow rates fall into ranges appropriate for high-volume (~1,000 L/min), medium-volume (~100 L/min), low-volume (~10 to 20 L/min) and mini-volume (<5 L/min) samplers. The medium- and high-volume inlets are especially useful when samples are taken in parallel on several substrates, since flow rates can be kept high enough to obtain an adequate deposit for analysis. Cut-points change with the inverse square root of the ratio of the actual flow to the cut-point flow. For example, decreasing the flow rate through the AIHL cyclone used in the IMPROVE sampler from 26.6 L/min to 20.6 L/min increases its d_{50} from 2.2 to 2.5 µm.

As Table 3-1 shows, tested inlets are available for high volume (~1,000 L/min), medium volume (~100 L/min), and low volume (~20 L/min) sampling with the most common cut-points being at 10 µm and from 2 to 3 µm. Several inlets can be placed in a series, in the form of a "cascade impactor," to obtain more detailed size distributions of chemical concentrations. Cascade impactors not listed in Table 3-1 are described by Hering (1995).

The principle of operation for each inlet is also identified in Table 3-1; these principles include direct impaction, virtual impaction, cyclonic flow, selective filtration, and elutriation (Marple et al., 1993). Impaction inlets (e.g., Olin and Bohn, 1983; McFarland et al., 1984; McFarland and Ortiz, 1984a-b) consist of circular or rectangular jets positioned above an impaction plate. The impactor dimensions are chosen such that particles smaller than the desired cut-point follow the streamlines as they bend at the impaction plate, while the larger particles with sufficient inertia depart from the streamlines and impact against the plate. Impactor design theory is highly developed (e.g., Wright, 1954; May and Clifford, 1967; Marple and Willeke, 1976a-b), and experimental sampling effectiveness curves generally agree with the theory (Marple and Rubow, 1986). To maintain their sampling effectiveness, particles must adhere to the impaction plate; particle re-entrainment and bounce of these large particles significantly degrades the performance of impaction inlets (Rao and Whitby, 1978a-b; Reischl and John, 1978). Surfaces can be oiled or greased to retain particles, and the impaction surfaces need to be regularly cleaned (Pitchford et al., 1997).

The $PM_{2.5}$ FRM WINS impactor is based on the principle of direct impaction. The replacement filter in the well of the WINS impactor needs to be changed after every 48 to 72 hours of sampling; the pre-inlet (modified SA 246B) preceding the WINS can be unscrewed for access to the impaction plate.

The virtual impactor used in the dichotomous sampler operates on a similar principle, with the exception that the impaction surface is replaced by an opening which directs the larger particles to one sampling substrate while the smaller particles follow the streamlines to another substrate. Particle bounce and re-entrainment are ameliorated by this method, but a fraction of the total flow (usually ~10%) must be drawn through the virtual impaction surface. A portion of the particles with diameters below the fine particle cut-point are collected with the larger particles and corrections must be made to the mass concentrations of these larger particles (Evans and Ryan, 1983).

Cyclonic flow inlets use an impeller to impart a circular motion to air entering the inlet (Chow and Lippman, 1977; McFarland et al., 1978; John and Reischl, 1980; Wedding et al., 1983; Wedding and Weigand, 1985). This air enters a cylindrical tube oriented perpendicular to the impellers and the centripetal force imparted to the particles in the airstream moves them toward the walls of this tube. Those particles reaching the tube wall either adhere to it, often with the help of an oil or grease coating, or drop into a “hopper” at the bottom of the collection tube. The hopper and inner tube are cleaned to minimize re-entrainment. Cyclones generally have a much higher loading capacity than impactor surfaces owing to the larger collection area and the reservoir provided by the hopper.

Selective filtration uses the uniform pore size and known sampling effectiveness of etched polycarbonate filters manufactured by Corning CoStar (formerly Nuclepore Corporation) to collect large particles on a pre-filter and pass smaller particles to a backup filter (John et al., 1983; Cahill et al., 1990). Microscopic examination shows that large particles are collected by impaction and interception around the pores. Eight micrometer pore size filters collect particles by interception and impaction in the vicinity of the pores to provide 50% cut-points for particles between 2 and 3 µm at flow rates of ~10 L/min. Cahill et al. (1990) observed re-entrainment of large, dry particles from the front filter and developed a greasing method to reduce this artifact.

Elutriator inlets draw air into a stilled-air chamber surrounding an open duct that leads to the filter. When the upward velocity due to flow through the inlet exceeds the particle settling velocity, that particle penetrates the inlet. When the settling velocity exceeds the upward velocity, the particle is not transmitted. This type of inlet was originally mated to the virtual impactor dichotomous sampler to provide a 15 µm cut-point. Wind tunnel tests (Olin, 1978; Wedding et al., 1980) found the cut-point to be highly dependent on wind speed, and this inlet was later replaced by the SA 246B, an impaction-type inlet.

3.1.2 Filter Media and Filter Holders

Particle sampling filters consist of a tightly woven fibrous mat or of a plastic membrane that has been penetrated by microscopic pores. Several air sampling filter types and manufacturers are identified in Table 3-2 with their physical and chemical characteristics and the laboratory analysis methods with which they are compatible. This table shows that no single filter medium is appropriate for all desired chemical analyses, and it is often necessary to sample on multiple substrates when chemical characterization is desired. Several characteristics are important to the selection of filter media for compliance measurements. They are:

Particle Sampling Efficiency*:* Filters should remove more than 99% of suspended particles drawn through them, regardless of particle size or flow rate. Lee and Ramamurthi (1993) and Lippmann (1995) tabulate the sampling efficiencies for several filter media with different pore sizes and flow rates. Cellulose-fiber filters and etched polycarbonate-membrane filters have efficiencies lower than 50% for some porosities, pore sizes, and particle sizes. Lower porosities and pore sizes generally result in higher sampling efficiencies. These characteristics also increase flow resistance, however.

Mechanical Stability*:* Filters should lie flat in the filter cassette inside of the sampler, remain in one piece, and provide a good seal with the sampling system to eliminate leaks. A brittle filter material may flake and negatively bias mass measurements. When different laboratory analyses require different sections of the sample, the filter must allow precise and accurate sectioning. The pure quartz-fiber filters listed in Table 3-2 are very brittle, and portions of their edges often become attached to the filter holder, thereby biasing the mass measurement. Ringed Teflon membranes are stretched between a ring, and these curl when they are cut in half or when a punch is removed.

Chemical Stability: Filters should not chemically react with the deposit, even when submitted to strong extraction solvents. They should not absorb gases that are not intended to be collected. When gas absorption is desired, as it may be when the precursors of secondary particles are measured, the filter material should absorb those gases at near 100% efficiency. In addition to the sulfate and nitrate absorption artifacts, Eatough et al. (1990) and McDow and Huntzicker (1990) demonstrate evidence of organic vapor adsorption on quartz-fiber filters. Demuynck (1975) and Charell and Hawley (1981) show the extent to which cellulose-fiber filters absorb water, and thereby bias mass measurements.

Temperature Stability: Filters should retain their porosity and structure in the presence of temperatures typical of the sampled airstream and of the analysis methods. All of the filters in Table 3-2 are stable at ambient temperatures, but they may melt when used near hot exhaust sources. Some analyses, such as those for carbon, heat the filter and its deposit to several hundred degrees Celsius, and a melted filter may encapsulate the deposit.

Blank Concentrations: Filters should not contain significant and highly variable concentrations of the chemicals which are being sought by analysis. Each batch of the unexposed filters should be examined for blank concentration levels prior to field sampling. These will be measured as if they were part of the particulate deposit.

Flow Resistance and Loading Capacity: Filters should allow sufficient amounts of air to be drawn through them to satisfy the flow rate requirements of the inlet and to obtain an adequate deposit. The collected particles should not clog them to the extent that flow rates decrease between sampling; particle concentrations up to 500 $\mu g/m^3$ should be attainable over a 24-hour period. Lee and Ramamurthi (1993) and Lippmann (1995) tabulate flow resistances for several types of filter. Membrane filters generally have higher flow resistances and lower loading capacities than fiber filters, and they are often used in samplers with low- and medium-volume inlets. Lower resistances and higher capacities can be attained by increasing the filter size, increasing the pore size, increasing the number of pores (in a membrane filter), and decreasing the filter thickness. Decreased flow resistance is often gained at the expense of decreased sampling efficiency.

Cost and Availability*:* Filters should be consistently manufactured and available at reasonable costs. The cost of filters varies substantially with filter material, filter size, the quantity purchased, and the current competition. Table 3-2 provides the current list price for various filters and the minimum number of filters per box. Ringed Teflon-membrane filters are typically the most costly (~$4.50 for each 47 mm diameter filter), with cellulose-fiber and glass-fiber filters (~$0.25 for each 47 mm diameter filter) being the least expensive. The cost of the filter is generally a small fraction of the cost of monitoring, and the validity of the measurement should not be compromised because one filter is "cheaper" than another. Filters are not always kept in stock, and even when they are, the lead times for acceptance testing and preparation can require one month or more. Filters should be procured well in advance of a monitoring program and in sufficient quantity to last the duration of the study.

To minimize contamination, filters can be loaded into and unloaded from filter holders in a clean laboratory environment rather than in the field. Watson and Chow (1993), Chow (1995), and Lippmann (1995) describe several different types of filter holders that are used in aerosol sampling, and some of this information is summarized in Table 3-3. Note that filter holders are made from a variety of materials (as are size-selective inlets) that must be considered when reactive components of suspended particles are measured.

Filter holders are configured as open-faced, with no constrictions upstream of the filter surface, or in-line, with a small diameter opening into a small chamber into which the filter is mounted. In-line holders often concentrate the particles in the center of the substrate, and this will bias the results if analyses are performed on portions of the filter. Tombach et al. (1987) and Fujita and Collins (1989) show differences as high as 600% between chemical measurements in the middle and at the edges of filters sampled with in-line filter holders. Open-faced filter holders are a better choice for ambient aerosol sampling systems. Exposed filters should not be subjected to excessive vibration that might dislodge the particles acquired on their surfaces (Dzubay and Barbour, 1983).

Many filters used in air sampling are manufactured for other chemical purposes (usually to remove contaminants rather than to collect them), and their properties must be verified with each procurement. Acceptance tests include: (1) examination of individual filters for holes, tears, and inhomogeneities; (2) batch verification of filter diameter and exposure area (especially for ringed Teflon filters; and (3) batch verification of blank levels for the species sought by chemical analysis.

3.1.3 Flow Measurement, Control, and Movement

As noted above, size-selective inlets require flow rates to be maintained within close tolerances to maintain the desired cut-point. Table 3-4 describes several of the flow measurement and control devices in common use for particle sampling. While manual flow control is adequate when filters do not load appreciably, most modern sampling devices use some form of feedback to adjust the pressure drop or pump speed to compensate for increasing flow resistance during sampling.

The distinction between volumetric and mass flow control is an important one. Inlet and inhalation characteristics depend on volume flow rates, not on the mass of the air transferred. Changes in temperature and pressure throughout a day and year cause mass and volumetric flow rates to differ. For this reason, FRM performance specifications require consistency of volumetric rather than mass flow rates. Wedding (1985) estimates potential differences in excess of 10% between mass and volumetric measurements of the same flow rates, depending on temperature and pressure variations. These differences can be minimized by re-calibrating mass flow controllers during each season at the median temperature for a typical sampling day during that season.

While critical orifices are simple and inexpensive, they require the downstream pressure to be less than ~50% of the pressure upstream of the orifice; this condition may not be attained for a heavily loaded filter that induces a high pressure drop across it, or for samplers operating at high altitudes. Critical orifices also require more massive pumps than other flow control devices, so the cost savings on flow control are offset by more expensive pump costs and maintenance. The critical throat design (Wedding et al., 1987) allows higher flow rates from a given pump by recovering some of the energy that is expended in back pressure behind a critical orifice.

Table 3-4 describes the principles of the pumps commonly used in particle samplers. Rubow and Furtado (1995) provide more detailed specifications for commercially available pumps. Pump capacities and power requirements must be matched to the flow resistance of the filters, the flow control method, inlet flow rate requirements, and available power. Some pumps are noisy, and the sound must be muffled when residents are near a sampling site (Sacco et al., 1976). Rogers et al. (1989) found that a 3/4 horsepower carbon vane pump is sufficient to draw in excess of 120 L/min through a 47 mm Teflon-membrane filter with 2 μm pore size. Smaller pumps can be used for lower flow rates and filter media with lower resistances. Pump capacity, filter media, flow controllers, and inlet flow requirements must be specifically matched for each sampling system.

3.2 Federal Reference and Equivalent Methods

Table 3-5 identifies the particle sampling systems that have achieved reference or equivalent status for PM_{10} compliance monitoring. $PM_{2.5}$ compliance monitoring networks (i.e., State and Local Air Monitoring Stations [SLAMS], National Ambient Monitoring Stations [NAMS]) are equipped with Federal Reference Method (FRM) and Federal Equivalent Method (FEM) samplers. IMPROVE samplers are located at regional transport and background sites to satisfy SLAMS requirements (U.S. Environmental Protection Agency, 1997a-b).

3.2.1 $PM_{2.5}$ Federal Reference Method

U.S. EPA (1997b) specifies sampler design, performance characteristics, and operational requirements applicable to the $PM_{2.5}$ FRM in 40 CFR part 50, Appendix L; 40 CFR part 53, Subpart E; and 40 CFR part 58, Appendix A (U.S. EPA, 1997a-d). $PM_{2.5}$ FRMs are intended to acquire deposits over 24-hour periods on Teflon-membrane filters from air drawn at a controlled flow rate through the WINS $PM_{2.5}$ inlet. The inlet and size separation components, filter types, filter cassettes, and internal configurations of the filter holder assemblies are specified by design, with drawings and manufacturing tolerances published in 40 CFR part 53 (U.S. EPA, 1997b). Other sampler components and procedures (such as flow rate control, operator interface controls, exterior housing, data acquisition) are specified by performance characteristics, with specific test methods to assess that performance.

Design specifications of the FRM samplers include the a modified SA-246 PM_{10} inlet (Figure 3-3) that has previously been wind tunnel tested and approved for PM_{10} compliance monitoring. The inlet cover has been extended by 2.5 inches and bent 45° downward to minimize water presentation during rainstorms. Sample air enters the inlet and is drawn through the WINS (Figure 3-4) that is designed to removes particles with aerodynamic diameter greater than 2.5 μm by impacting them on the bottom of an open-topped aluminum cylindrical container. The impacting particles are trapped at the bottom of the well on an oil-impregnated filter (35 to 37 mm borosilicate glass-fiber) impregnated with a low vapor-pressure oil (tetramethyltetraphenyltrisiloxane, maximum vapor pressure 2×10^{-8} mm Hg, density 1.06 to 1.07 g/cm^3, 32 to 40 centistoke viscosity at 25 °C). More than 50% of the particles with aerodynamic diameters less than 2.5 μm follow the air flow through the WINS, which turns up and out of the well and is directed back down to a Teflon-membrane filter where the particles are removed by filtration. The fraction of each particle size passing through the WINS is shown in Figure 3-1. Internal surfaces exposed to sample air prior to the Teflon-membrane filter are treated electrolytically in a sulfuric acid bath to produce a clear, uniformly anodized coating (at least 1.08 mg/cm^2 in accordance with military standard specifications).

Several $PM_{2.5}$ samplers using the WINS and other $PM_{2.5}$ inlets have been operated simultaneously in Birmingham, AL; Denver, CO; Phoenix, AZ; Tucson, AZ; Bakersfield, CA; and Azusa, CA; from November 1996 through May 1997 (Pitchford et al., 1997). Figure 3-5 (Pitchford et al., 1997) compares $PM_{2.5}$ measurements from two WINS samplers with simultaneous measurements from dichotomous and IMPROVE samplers. This comparison and others from areas with different particle size distributions and compositions show a reasonable equivalence between WINS and other $PM_{2.5}$ inlets, as well as collocated precisions of ~0.5 to 1.0 μg/m^3 among WINS samplers.

FRM performance specifications require constant volumetric flow rates (16.67 ± 0.83 L/min) to be monitored and recorded continuously with temperature and pressure of the sample air entering the inlet and near the filter. FRMs are required to maintain the temperature of the filter during and after sampling within ±5 °C of concurrent ambient temperatures regardless of heating and cooling from direct sun or shade during and after sampling. This specification intends to minimize losses from volatile particles such as ammonium nitrate and some organic compounds. Potential FRM designs use active ventilation of the enclosure that surrounds the filter holder and WINS impactor to attain these temperature performance specifications.

FRMs from different manufacturers may vary in appearance, but their principles of operation and resulting $PM_{2.5}$ mass measurements should be the same within reasonable measurement precisions. Though they may follow the published design specifications, $PM_{2.5}$ samplers are not FRMs until they have demonstrated attainment of the published specifications

(U.S. EPA, 1997b) and assigned an FRM number published in the Federal Register.

3.2.2 Class I $PM_{2.5}$ Federal Equivalent Method

Federal Equivalent Methods (FEMs) are divided into several classes in order to encourage innovation and provide monitoring flexibility. This is especially important for chemical characterization, as more than one filter medium is required to account for the significant chemical components.

Class I FEMs meet nearly all FRM specifications, with minor design changes that permit sequential sampling without operator intervention and different filter media in parallel or in series. Flow rate, inlets, and temperature requirements are identical for FRMs and Class I FEMs. Particles losses in flow diversion tubes are to be quantified and must be in compliance with Class I FEM tolerances specified in 40 CFR part 53, Subpart E.

3.2.3 Class II $PM_{2.5}$ Federal Equivalent Method

Class II FEMs include samplers that acquire 24-hour integrated filter deposits for gravimetric analysis, but that differ substantially in design from the reference-method instruments. These might include dichotomous samplers, high-volume samplers with $PM_{2.5}$ size-selective inlets, and other samplers identified in Table 3-6. More extensive performance testing is required for Class II FEMs than for FRMs or Class I FEMs, as described in 40 CFR part 53, Subpart F. Key requirements for Class I and Class II FEM equivalence tests are summarized in Table 3-7.

3.2.4 Class III $PM_{2.5}$ Federal Equivalent Method

Class III FEMs include samplers that do not qualify as Class I or Class II FEMS. This category is intended to encourage the development of and to evaluate new monitoring technologies that increase the specificity of $PM_{2.5}$ measurements or decrease the costs of acquiring a large number of measurements. Class III FEMs may either be filter-based integrated samplers or filter- or non-filter-based *in situ* continuous or semi-continuous samplers. Table 3-8 identifies several types of continuous particle measurement instruments may be candidates for Class III FEM status.

Watson et al. (1997b) describes these instruments in greater detail and describes current knowledge about how well they approximate $PM_{2.5}$ concentrations under a variety of environmental conditions. Test procedures and performance requirements for Class III candidate method instruments will be determined on a case-by-case basis. Performance criteria for Class III FEMs will be the most restrictive, because equivalency to reference methods must be demonstrated over a wide range of particle size distributions and aerosol compositions.

3.3 IMPROVE Samplers

IMPROVE (Interagency Monitoring of PRotected Visual Environments) samplers can be used at regional background and transport sites to fulfill SLAMS requirements. IMPROVE samplers were developed for the IMPROVE network (~70 locations shown in Figure 3-6) to quantify PM chemical components that affect visibility at Federal Class I areas that include National Parks, National Monuments, and Wilderness Areas.

IMPROVE samplers consist of up to four parallel filter and inlet combinations (Modules A, B, and C for $PM_{2.5}$; Module D for PM_{10}) controlled by a common timer. All IMPROVE sites have module A to determine $PM_{2.5}$ mass and elemental composition, and additional modules are added when other size fractions and chemical components are desired. Each of the three $PM_{2.5}$ modules utilizes a modified Air Industrial Hygiene Laboratory (AIHL) cyclone as a $PM_{2.5}$ inlet at a flow rate of 23 L/min, a 25 or 47 mm Nuclepore filter holder assembly, a volumetric flow control device, and a pump (Eldred et al., 1988, 1990).

Module A uses a Teflon-membrane filter to measure $PM_{2.5}$ mass by gravimetry (Watson et al., 1995b), light absorption (b_{abs}) by the integrated plate method (Campbell et al., 1989), hydrogen by proton elastic scattering analysis (PESA) (Cahill et al., 1971), and elements (Na to Pb) by proton induced x-ray emission analysis (PIXE) (Cahill, 1985). Module B is equipped with an acid-vapor diffusion denuder followed by a nylon filter to measure total (non-volatilized and volatilized) particulate nitrate by ion chromatography (Chow and Watson, 1997c). Module C contains two pre-fired quartz-fiber filters in series to measure organic and elemental carbon on the front filter and to assess the extent of organic artifacts on the backup filter by thermal/optical reflectance analysis (TOR) (Chow et al., 1993b).

To examine the magnitude of nitrate volatilization, Module B can be modified to adapt tandem nylon-membrane or sodium-chloride-impregnated cellulose-fiber filters to measure non-volatilized particulate nitrate on the front filter and volatilized particulate nitrate on the backup filter.

3.4 Research Samplers

Research measurements at Special Monitoring Sites (SPMs) may require other particle samplers that are not classified as FRMs or FEMs. These may be needed and useful for understanding excessive $PM_{2.5}$ concentrations measured by FRMs and FEMs at compliance sites, but they are not eligible for determining compliance with the $PM_{2.5}$ NAAQS. Such instruments are not precluded from becoming equivalent on a site-specific, regional, or national basis, but such a designation is not required when these instruments are used to apply advanced sampling and laboratory analysis techniques that help to advance understanding of source contributions and health effects..

Table 3-6 summarizes the characteristics of several research sampling systems that have been assembled from the basic sampling components and applied in field studies. Many of these include several inlets, denuders, filters in series and parallel, and different absorbing materials to accurately measure volatile compounds and to quantify the precursors of secondary aerosols. Knowledge of research monitors is important since some of these research monitors may become future compliance monitors. Well-characterized size-selective inlets and filter media might not be available at affordable costs without the large volume of sales engendered by compliance monitoring networks. Watson and Chow (1993, 1994c) describe several of these samplers in greater detail and cite references to the research studies in which they were used.

Many of the sampling systems in Table 3-6 are complex, involve numerous sampling substrates and laboratory analyses, and require substantial skill and dedication to operate. These characteristics are not entirely compatible with the needs of compliance monitoring networks where budgets are limited and field technicians have many duties other than maintaining particle monitors. Nevertheless, Table 3-6 does indicate some directions that might be considered for monitoring compliance with new air quality standards.

The sequential filter sampler (SFS) and California Acid Deposition Monitoring Program (CADMP) dry deposition samplers (Chow et al., 1993b) listed in Table 3-6 are offspring of the sequential filter sampler PM_{10} reference method (RFPS-0389-071, see Table 3-5). These medium-volume samplers draw air through medium-volume inlets into a plenum. Several samples can then be drawn simultaneously from the plenum, through denuders, and onto various filter media if necessary. By adjusting a make-up flow rate, flow remains constant as long as the air drawn through all filters does not exceed the flow needed for the specified cut-point. The SFS is especially applicable to samples of less than 24 hours when flow rates are increased to compensate for shorter sample durations (Chow and Egami, 1997).

The last sampler in Table 3-6, the Minivol Portable Survey Sampler, is a development in the direction of simplicity rather than complexity. This inexpensive (~$2,000) battery- or solar-powered unit can be hung from power poles and building walls and does not require complicated sampler siting, security, or power sources. It can be placed in and around fixed sampling sites to determine how well these sites represent community exposure. It can also be located within and around areas of source emissions to quantify the "zones of influence" of these emissions. Though its flow rate is low and its inlet is minimally characterized, Chow and Watson (1997b) found good agreement between 24-hour average PM_{10} mass measurements from the Minivol with collocated measurements from a hivol-SSI, a sequential filter sampler, a dichotomous sampler, and a beta attenuation monitor.

Pitchford et al. (1997) reported reasonable agreement between FRM and portable $PM_{2.5}$ survey samplers in Phoenix, AZ. The trade-offs in accuracy and precision of a single measurement need to be balanced against the better spatial representation and improved assessment of human exposure that these samplers can provide.

Table 3-1
Size-Selective Inlets for Aerosol Sampling

Inlet Identifier (Manufacturer)	References	Operating Principle	d_{50} (µm); Slope (no unit)	Flow Rate (L/min)	PM_{10} Reference or Equivalence Reference No.[a]	Comments
High-Volume						
SA[a] or GMC[c] Model 320 (Graseby Andersen, Atlanta, GA)	McFarland et al. (1980)	Impactor	15; 1.5	1,133	None	Single-stage, no greased shim.
SA or GMW Model 321A	McFarland and Ortiz (1984); Hayes et al. (1988)	Impactor	10.2; 1.45	1,133	RFPS-1287-065	Two-stage with greased shim.
SA or GMW Model 321B	Hayes et al. (1988); McFarland and Ortiz (1987)	Impactor	9.7; 1.40	1,133	RFPS-1287-064	Two-stage with greased shim.
SA or GMW Model 1200	Purdue (1988); Wedding et al. (1988); Mathai et al. (1988); Hoffman et al. (1988); Hayes et al. (1988)	Impactor	9.7; 1.40	1,133	RFPS-1287-063	Single-stage with greased shim (body hinged).
GMW Wedding PM_{10} (General Metal Works, Village of Cleves, OH)	Woods et al. (1986)	Cyclonic Flow	8.8;	1,133	None	No inlet cleaning port.
Wedding IP_{10} PM_{10} (Wedding & Associates, Fort Collins, CO)	Wedding and Weigand (1985); Woods et al. (1986)	Cyclonic Flow	9.6; 1.37	1,133	RFPS-1087-062	Inlet cleaning port on top of inlet.

Table 3-1 (continued)
Size-Selective Inlets for Aerosol Sampling

Inlet Identifier (Manufacturer)	References	Operating Principle	d_{50} (µm); Slope (no unit)	Flow Rate (L/min)	PM_{10} Reference or Equivalence Reference No.[a]	Comments
Medium-Volume						
SA 254 Medium-Volume PM_{10} Inlet	Olin and Bohn (1983)	Impactor	10; 1.6[d]	113	RFPS-0389-071	Several small screws must be removed to clean.
Wedding Medium Flow PM_{10} Inlet	Wedding et al. (1983)	Cyclonic Flow	9.5; 1.12	113	None	Has a cleaning port. Can use a bottle brush to clean.
Bendix 240 Cyclone (Sensidyne, Inc., Clearwater, FL)	Chan and Lippmann (1977); Mueller et al. (1983)	Cyclonic Flow	2.5; 1.7	113	None	Plastic cap acts as a hopper to collect large particles.
Low-Volume						
SA 246B	McFarland and Ortiz (1984); Van Osdell and Chen (1990)	Virtual Impactor	10.2; 1.41	16.7	RFPS-0789-073, EQPM-0990-076	Top unscrews to allow access to impaction surface.
Sierra-Anderson 244 and 245	McFarland et al. (1978); Olin (1978)	Virtual Impactor	2.5;	16.7	None	Virtual impactor can be re-assembled backwards when taken apart for cleaning.
AirMetrics Inc. (Springfield, OR)	Chow and Watson (1996)	Direct Impactor	10; 2.5;	5 5	None	Nylon impactor needs to be cleaned and regreased after every 72 hours of sampling.

Table 3-1 (continued)
Size-Selective Inlets for Aerosol Sampling

Inlet Identifier (Manufacturer)	References	Operating Principle	d_{50} (µm); Slope (no unit)	Flow Rate (L/min)	PM_{10} Reference or Equivalence Reference No.[a]	Comments
Low-Volume (continued)						
WINS (Well Impactor – Ninety Six)	U.S. EPA (1997b)	Direct Impactor	2.5; 1.18[d]	16.7	None	Inlet used in $PM_{2.5}$ FRM.
USEPA/HEAD Impactor	Koutrakis et al. (1990)	Direct Impactor	2.1; 1.08[d]	10	None	Oiled glass impactor needs to be replaced after every 24 hours of sampling.
Wedding TP_{10}	Wedding et al. (1982)	Cyclonic Flow	9.9; 1.32[d]	16.7	EQPM-0391-081	Plastic cap acts as a hopper to collect large particles.
Bendix Unico 18	Chan and Lippmann (1977)	Cyclonic Flow	2.5; 1.83[d]	18	None	
AIHL Cyclone (Air & Industrial Hygiene Laboratory, Berkeley, CA)	John and Reischl (1980)	Cyclonic Flow	2.2; 1.87[e] 2.5; 1.38[e] 3.5; 1.40[e]	26.6 21.7 15.4	None	Screw-on cap acts as a hopper to collect large particles.
Stacked Filter Unit (Corning CoStar [formerly Nuclepore Corp.], Cambridge, MA)	Flocchini et al. (1981); John et al. (1983); Cahill et al. (1990)	Selective Filtration	2 to 3;	10	None	Uses large pore (8 µm) etched polycarbonate filters.
BGI-4 (BGI Inc., Waltham, MA)	Blackman and Lippmann (1974); Hering (1995)	Cyclonic Flow	4;	2.3	None	Generally used in personal sampling applications.

Table 3-1 (continued)
Size-Selective Inlets for Aerosol Sampling

Inlet Identifier (Manufacturer)	References	Operating Principle	d_{50} (µm); Slope (no unit)	Flow Rate (L/min)	PM_{10} Reference or Equivalence Reference No.[a]	Comments
Low-Volume (continued)						
MSA (Mine Safety Appliances Co., Pittsburgh, PA)	Blackman and Lippmann (1974); Hering (1995)	Cyclonic Flow	3.5;	2	None	Generally used in personal sampling applications.
Sensidyne Model BDX 99R (Sensidyne Inc., Clearwater, FL)	Blackman and Lippmann (1974); Hering (1995)	Cyclonic Flow	3.5;	1.7	None	Also known as Dorr-Oliver design. Generally used in personal sampling applications.
SKC Cat. No. 225-01-02 (SKC Inc., Eighty Four, PA)	Blackman and Lippmann (1974); Hering (1995)	Cyclonic Flow	5;	1.9	None	Generally used in personal sampling applications.
MST Low Flow Rate Sharp Cut Impactor (Harvard Impactor) (Air Diagnostic and Engineering, Harrison, ME)	Marple et al. (1987)	Direct Impactor	2.5; 1.02[d] 10; 1.11[d]	4, 10, 20 4, 10	None None	Oiled aluminum impactor plate needs to be replaced after every 24 hours of sampling. Designed for use in indoor air pollution health studies.

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[a] See Table 3-5 for Federal Register citation and notice date.

[b] Sierra-Andersen, Atlanta, GA.

[c] General Metal Works, Atlanta, GA.

[d] Slope = $\sqrt{d_{16} / d_{84}}$, as defined in text.

[e] Slope is estimated based on $\sqrt{d_{10} / d_{90}}$.

[f] Inlet for Modules A, B, and C of IMPROVE samplers.

Table 3-2

Commonly Used Filter Media for Particulate Sampling and Analysis

Filter Type, (Major Manufacturer, and Catalog No. or Trade Name)	Filter Size	Physical Characteristics	Chemical Characteristics	Compatible Analysis Methods[a]	Cost per Filter	No. of Filters per Box
• Ringed Teflon-membrane (Gelman Scientific; Ann Arbor, MI; Teflo®, R2PJ047, R2PJ037)	25 mm 37 mm 47 mm	• Thin membrane stretched between polymethylpentane ring. • White surface, nearly transparent. • Minimal diffusion of transmitted light. • High particle collection efficiencies. • Cannot be accurately sectioned. • 1.2, 2.0, 3.0, 5.0 and 10 µm pore sizes (determined from liquid filtration). • Melts at ~60°C. • High flow resistance.	• Usually low blank levels, but several contaminated batches have been found. Made of carbon-based material, so inappropriate for carbon analysis. • Inert to adsorption of gases. • Low hygroscopicity. • Low blank weight.	• Gravimetry, OA, XRF, PIXE, INAA, AAS, ICP/AES, ICP/MS, IC, AC	$3.78 $4.04 $4.38	50 50 50
• Ringed Teflon-membrane (Pallflex, Putnam, CT)	25 mm 37 mm 47 mm	• Thin film of Teflon attached to polyolefin ring without adhesive.	• Made of carbon-based material, so inappropriate for carbon analysis. • Inert to adsorption of gases. • Low hygroscopicity. • Low blank weight.	• Gravimetry, OA, XRF, PIXE, INAA, AAS, ICP/AES, ICP/MS, IC, AC	 $2.36 $2.50	 25 25

Table 3-2 (continued)
Commonly Used Filter Media for Particulate Sampling and Analysis

Filter Type, (Major Manufacturer, and Catalog No. or Trade Name)	Filter Size	Physical Characteristics	Chemical Characteristics	Compatible Analysis Methods[a]	Cost per Filter	No. of Filters per Box
• Backed Teflon membrane, (Gelman Scientific, Ann Arbor, MI; "Zefluor"; 2 µm, P5PJ037 or P5PJ047; 1 µm, P5PL037 or P5PL047)	37 mm 47 mm 20.3 × 25.4 cm	• Thin membrane mounted on thick polypropylene backing. • White opaque surface, diffuses transmitted light. • High particle collection efficiencies. • Melts at ~60°C. • High flow resistance. • 1 µm and 2 µm pore sizes.	• Usually low blank levels. Made of carbon-based material, so inappropriate for carbon analysis. • Inert to adsorption of gases. • Higher background levels for XRF and PIXE than Teflo® owing to greater filter thickness. • Low hygroscopicity. • High blank weight.	• Gravimetry, XRF, PIXE, INAA, AAS, ICP/AES, ICP/MS, IC, AC	$1.98 $2.30 $29.40	50 50 25
• Backed Teflon membrane, (Pallflex, Putnam, CT)	25 mm 37 mm 47 mm	• TFE porous membrane on TFE support. • Smooth surface. • 0.30 µm @ 99% efficiency.	• Neutral pH. • carbon-based material, so inappropriate for carbon analysis. • Inert to adsorption of gases. • Higher background levels for XRF and PIXE than Teflo® owing to greater filter thickness. • Low hygroscopicity. • High blank weight. • Retains average tare weight of 7.6 grams.	• Gravimetry, XRF, PIXE, INAA, AAS, ICP/AES, ICP/MS, IC, AC	 $2.14 $2.57	 50 50

Table 3-2 (continued)
Commonly Used Filter Media for Particulate Sampling and Analysis

Filter Type, (Major Manufacturer, and Catalog No. or Trade Name)	Filter Size	Physical Characteristics	Chemical Characteristics	Compatible Analysis Methods[a]	Cost per Filter	No. of Filters per Box
• Nylon membrane, (Gelman Scientific, Ann Arbor, MI; "Nylasorb", #66509)	47 mm 90 mm	• Thin membrane of pure nylon. • White opaque surface, diffuses transmitted light. • 1 µm pore size. • Melts at ~60°C. • High flow resistance.	• High HNO_3 collection efficiency. • Passively adsorbs low levels of NO, NO_2, PAN, and SO_2. • Low hygroscopicity. • Low blank weight.	• IC, AC	$1.70 $5.00	100 50
• Silver membrane (Millipore Corp., Marlborough, MA)	25 mm[b] 37 mm[c]	• Thin membrane of sintering, uniform metallic silver particles. • Grayish-white surface diffuses transmitted light. • Melts at ~350°C. High flow resistance.	• Resistant to chemical attack by all fluids. • Passively adsorbs organic vapors. • Low hygroscopicity. • High blank weight.	• Gravimetry, XRD	$2.71 $3.88	50 25
• Cellulose esters membrane (Millipore Corp., Marlborough, MA;" Nitrocellulose")	37 mm 47 mm[d]	• Thin membrane of cellulose nitrate mixed esters, and cellulose acetate. • White opaque surface diffuses transmitted light. • 0.025, 0.05, 0.1, 0.22, 0.30, 0.45, 0.65, 0.80, 1.2, 3.0, 5.0, and 8.0 µm pore sizes. • Melts at ~70°C. • High flow resistance.	• High hygroscopicity. • Negligible ash content. • Dissolves in many organic solvents. • Low hygroscopicity. • Low blank weight.	• Gravimetry, OM, TEM, SEM, XRD • Biomedical applications	$0.70 $0.67	100 100

Table 3-2 (continued)
Commonly Used Filter Media for Particulate Sampling and Analysis

Filter Type, (Major Manufacturer, and Catalog No. or Trade Name)	Filter Size	Physical Characteristics	Chemical Characteristics	Compatible Analysis Methods[a]	Cost per Filter	No. of Filters per Box
• Polyvinyl Chloride membrane (Millipore Corp., Marlborough, MA).	25 mm 37 mm 47 mm	• Hospital-grade polyvinyl chloride membrane. • White opaque surface, diffuses transmitted light. • 0.2, 0.6, 0.8,2.0, and 5.0 µm pore sizes. • Melts at ~50°C. • High flow resistance.	• Dissolves in some organic solvents. • High hygroscopicity. • Low blank weight.	• XRD	$0.76 $1.03 $1.19	100 100 100
• Polycarbonate membrane, (Corning CoStar, [formerly Nuclepore Corp.], Cambridge, MA; #111129) (Poretics, Minnetonka, MN)	25 mm 37 mm 47 mm[b]	• Smooth, thin, polycarbonate surface with straight through capillary holes. • Used for particle size classification. • Light gray surface, nearly transparent. • Minimal diffusion of transmitted light. • Low particle collection efficiencies, <70% for some larger pore sizes. • Retains static charge. • 0.1, 0.3, 0.4, 0.6, 1.0, 2.0, 3.0, 5.0, 8.0, 10.0, and 12.0 µm uniform pore sizes. • Melts at ~60°C. • Moderate flow resistance.	• Low blank levels (made of carbon-based material, so inappropriate for carbon analysis). • Low hygroscopicity. • Low blank weight.	• Gravimetry, OA, OM, SEM, XRF, PIXE	$0.32 $0.44 $0.51	100 100 100

Table 3-2 (continued)
Commonly Used Filter Media for Particulate Sampling and Analysis

Filter Type, (Major Manufacturer, and Catalog No. or Trade Name)	Filter Size	Physical Characteristics	Chemical Characteristics	Compatible Analysis Methods[a]	Cost per Filter	No. of Filters per Box
• Pure quartz-fiber (Pallflex Corp., Putnam, CT; 2500 QAT-UP)	25 mm 37 mm 47 mm 20.3 × 25.4 cm	• Mat of pure quartz fibers. • White opaque surface, diffuses transmitted light. • High particle collection efficiencies. • Soft and friable edges flake in most filter holders. • Melts at >900°C. • Moderate flow resistance.	• Pre-washed during manufacture-low blank levels for ions. • Contains large and variable quantities of Al and Si. Some batches contain other metals. • Passively adsorbs organic vapors. Adsorbs little HNO_3, NO_2, and SO_2. • Low hygroscopicity.	• ICP/AES, ICP/MS, IC, AC, T, TOR, TMO,TOT, OA	\$1.10 \$0.62 \$0.50 \$6.70	100 25 25 25
• Mixed quartz-fiber (Whatman Corp., Hillsboro, OR; QM/A #1861865)	37 mm 47 mm 20.3 × 25.4 cm	• Quartz (SiO_2) fibers with ~5% borosilicate content. • White opaque surface, diffuses transmitted light. • High particle collection efficiencies. • Some batches can melt at ~500°C. Effects on thermal carbon analysis are unknown. • Becomes brittle when heated. • Low flow resistance.	• High blank weight. • Contains large and variable quantities of Na, Al, and Si in all batches. Variable levels of other metals are found in many batches. • Passively adsorbs organic vapors. Adsorbs little HNO_3, NO_2, and SO_2. • Low hygroscopicity. • High blank weight.	• Gravimetry, XRF, PIXE, AA, ICP/AES, ICP/MS for some metals, IC, AC, T, TOR, TMO, TOT	\$0.67 \$0.74 \$6.40	100 100 25

Table 3-2 (continued)
Commonly Used Filter Media for Particulate Sampling and Analysis

Filter Type, (Major Manufacturer, and Catalog No. or Trade Name)	Filter Size	Physical Characteristics	Chemical Characteristics	Compatible Analysis Methods[a]	Cost per Filter	No. of Filters per Box
• Cellulose-fiber 41 (Whatman Corp., Hillsboro, OR; #1441047)	25 mm 47 mm	• Thick mat of cellulose fibers, often called a "paper" filter. • White opaque surface, diffuses transmitted light. • Low particle collection efficiencies, <70% for some variations of this filter. • High mechanical strength. • Burns at elevated temperatures (~150°C, exact temperature depends on nature of particle deposit). • Variable flow resistance.	• High purity, low blank levels. Made of carbon-based material, so inappropriate for carbon analysis. • Adsorbs gases, especially water vapor. • Most appropriate for adsorbing gases such as HNO_3, SO_2, NH_3, and NO_2 when impregnated with reactive chemicals. • High hygroscopicity. • High blank weight.	• Gravimetry, XRF, PIXE, INAA, AAS, ICP/AES, ICP/MS, IC, AC	\$0.10 \$1.05	100 100
• Cellulose-fiber 31ET (Whatman Corp., Hillsboro, OR; #3031F915)	47 mm 46 × 57 cm	• 0.5 mm thick. • Less flow resistance than Whatman 41.	• High purity, low blank levels. Made of carbon-based material, so inappropriate for carbon analysis. • Adsorbs gases, especially water vapor. • Most appropriate for adsorbing gases such as HNO_3, SO_2, NH_3, and NO_2 when impregnated with reactive chemicals. • High hygroscopicity. • High blank weight.	• Gravimetry, XRF, PIXE, INAA, AAS, ICP/AES, ICP/MS, IC, AC	\$0.17[e] \$7.28	100 25

Table 3-2 (continued)
Commonly Used Filter Media for Particulate Sampling and Analysis

Filter Type, (Major Manufacturer, and Catalog No. or Trade Name)	Filter Size	Physical Characteristics	Chemical Characteristics	Compatible Analysis Methods[a]	Cost per Filter	No. of Filters per Box
• Teflon-coated glass-fiber (Pallflex, Putnam, CT; TX40HI20)	37 mm 47 mm	• Thick mat of borosilicate glass fiber with a layer of Teflon on the surface. • Glass fiber supporting Teflon is shiny. • High particle collection efficiencies. • Glass melts at ~500°C. Teflon melts at ~60°C. • Low flow resistance.	• Low blank levels for ions (glass backing and carbon content make it less suitable for elemental and carbon analyses). • Inert to adsorption of HNO_3, NO_2, and SO_2. • Low hygroscopicity. • High blank weight.	• Gravimetry, IC, AC	$0.60 $0.63	100 100
• Glass fiber (Gelman Scientific, Ann Arbor, MI; Type A/E)	25 mm 47 mm 20.3 x 25.4 cm (available in 13 mm to 293 mm sizes)	• Borosilicate glass fiber. • White opaque surface, diffuses transmitted light. • High particle collection efficiencies. • Melts at ~500°C. Low flow resistance.	• High blank levels. • Adsorbs HNO_3, NO_2, SO_2, and organic vapors. • Low hygroscopicity. • High blank weight.	• Gravimetry, OA, XRF, PIXE, INAA, AAS, ICP/AES, IC, AC	$0.14 $2.40 $12.90	500 100 100

Table 3-2 (continued)
Commonly Used Filter Media for Particulate Sampling and Analysis

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[a] AAS = Atomic Absorption Spectrophotometry
AC = Automated Colorimetry
IC = Ion Chromatography
ICP/AES = Inductively-Coupled Plasma with Atomic Emission Spectrophotometry
ICP/MS = Inductively-Coupled Plasma with Mass Spectrophotometry
INAA = Instrumental Neutron Activation Analysis
OA = Optical Absorption or Light Transmission (b_{abs})
OM = Optical Microscopy
PIXE = Proton-Induced X-Ray Emissions
SEM = Scanning Electron Microscopy
T = Thermal Carbon Analysis
TEM = Transmission Electron Microscopy
TMO = Thermal Manganese Oxidation Carbon Analysis
TOR = Thermal/Optical Reflectance Carbon Analysis
TOT = Thermal Optical Transmission Carbon Analysis
XRD = X-Ray Diffraction
XRF = X-Ray Fluorescence

[b] Available in 0.45 µm pore size.

[c] Available in 0.80 µm pore size.

[d] Filter disc is available in-size between 13 mm to 293 mm depending on the pore size.

[e] Available by special order.

Table 3-3
Filter Holders and Their Characteristics

Type of Filter Holders (Manufacturer)	Filter Size	Physical Characteristics	Comments
• **Polycarbonate** (Corning Costar Corporation [Formerly Nuclepore] Cambridge, MA)	25 mm 47 mm	• In-line or open-face. • Polycarbonate base with flow resistant outlet. • Polycarbonate support grid with ethylene O-ring. • Extender section for multi-stage filter pack sampling.	• Use open-face holders for homogeneous filter deposit. • Need to bore the outlet to reduce flow resistance and fit to 3/8 inches of hose barb. • Use Viton O-ring to minimize organic sampling artifact. • Place resistance-free drain disk behind the filter medium to ensure filter integrity and prevent leakage.
• **Polypropylene** (Millipore Corp. Marlborough, MA)	13 mm 25 mm 47 mm	• In-line[a]. • Polypropylene or glass-filled polystyrene[b] base. • Polypropylene support grid with silicon O-ring.	• Used for occupational health applications.
(Graseby-Andersen, Inc. Atlanta, GA)	37 mm	• Open-face. • Polypropylene base.	
• **Polysulfone** (Schleicher and Schuell Inc., Keene, NH)	47 mm 50 mm	• Open face • Polysulfone fittings and support.	

Table 3-3 (continued)
Filter Holders and Their Characteristics

Type of Filter Holders (Manufacturer)	Filter Size	Physical Characteristics	Comments
• **Aluminum or Stainless Steel** (Gelman Instrument Co. Ann Arbor, MI)	25 mm 37 mm 47 mm	• In-line or open-face. • Stainless steel screen with Viton O-ring. • Nylon or polyethylene adapters.	• Used in industrial hygiene and occupational health applications.
(Millipore Corp. Marlborough, MA)	25 mm 47 mm	• In-line or open-face. • Stainless steel base. • Stainless steel screen with Teflon O-ring.	
(Graseby-Andersen, Inc. Atlanta, GA)	47 mm 20.3 x 25.4 cm	• In-line or open-face. • Stainless steel base. • Stainless steel screen with viton O-ring.	
(BGI Inc. Waltham, MA)	47 mm	• In-line or open-face. • Stainless steel base • Stainless steel or nickel-plated brass screen with silicon O-ring. • Stainless steel or nickel-plated brass adapter.	
• **PFA[c] Teflon** (Savillex Corp. Minnetonka, MN)	47 mm	• In-line or open-face. • PFA Teflon base injection molded. • PFA Teflon support grid with Viton O-ring. • PFA Teflon adapter.	• PFA Teflon is inert to gases such as HNO_3, NO_2, and SO_2. It doesn't remove them. • Use up to three multiple support grids for filter pack sampling.

Table 3-3 (continued)
Filter Holders and Their Characteristics

Type of Filter Holders (Manufacturer)	Filter Size	Physical Characteristics	Comments
• **Delrin** (Gelman Instrument Co., Ann Arbor, MI)	25 mm	• Open face. • Stainless steel support. • Nylon fittings.	
• **Teflon** (University Research Glassware Canboro, NC)	47 mm	• In-line. • Teflon base. • Teflon-coated stainless steel support grid[c].	• Available in double-stage Teflon-coated stainless steel.

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[a] 25 mm open-face is available of polypropylene base and cellulose pad support grid.
[b] Only available for 47 mm.
[c] PFA = Perfluoralkoxy.

Table 3-4
Flow Measurement, Flow Control, and Flow Movers

	Device and Flow Range	Principle of Operation	Comments
I.	***Flow Measurement***		
	Spirometer (Nelson, 1976)	The gas flow is directed into an inverted bell or cylinder of accurately known volume immersed in a liquid; the bell or cylinder is counterweighted, allowing the gas flow to raise it above the liquid surface with negligible resistance.	Primary standard; the vapor pressure of the liquid must be taken into account, as it effectively adds gas volume to the incoming flow.
	Pitot Tube (Hinds, 1982)	The ram (Bernoulli Effect) pressure of a gas stream is measured by an axial orifice and converted to flow rate, after subtracting the gas static pressure.	Primary standard; the velocity profile of the gas stream must be measured or assumed in order to convert the ram pressure velocity measurement into an integrated flow rate.
	Frictionless Pistons (Chen, 1993)	The gas flow raises a sealed but low-friction piston (e.g., a soap film) in a calibrated cylinder. The displaced volume is accurately known based on the dimensions of the cylinder.	Primary standard; soap-film versions require correction for the vapor pressure of the aqueous soap solution, and are subject to gas permeation of the film at higher flow rates. Mercury-sealed pistons require a correction for the weight of the piston.
	Aspirator Bottles (Nelson, 1976)	The gas stream displaces water or another fluid from a sealed bottle into a calibrated receptacle (e.g., graduated cylinder). The displaced volume is accurately measured in the receptacle, with a known filling time.	Primary standard; mostly limited to very small gas flows. If water is the displaced fluid, a vapor pressure correction is necessary.

Table 3-4 (continued)
Flow Measurement, Flow Control, and Flow Movers

Device and Flow Range	Principle of Operation	Comments
Dry Gas Meter (Hinds, 1982)	Volumetric flow rate is measured directly by the alternating filling and emptying of two bellows in a shared rigid enclosure.	Transfer standard; usually not appropriate for very low flow rate measurements.
Wet Test Meter (Lippmann, 1995)	Positive displacement of vanes partially immersed in a fluid (usually water).	Transfer standard; air or other gases may dissolve in or react with fluid.
Calibrated Rotameter (Okladek, 1988; Lippmann, 1995)	Height of ball or float in a tapered tube is proportional to volumetric flow rate.	Transfer standard; calibration curve must be corrected for density of air at flowmeter inlet.
Calibrated Mass Flow Meter (Wedding, 1985)	Mass flow rate is measured by sensing heat transfer from a heated element exposed to the flow.	Transfer standard; ambient air temperature fluctuations affect air density and mass flow rate, hence correction to volumetric flow rate is necessary.
Calibrated Orifice (Lippmann, 1995)	Pressure drop across a precisely-machined orifice is proportional to volumetric flow rate.	Transfer standard; simple design without any sensor or float inserted into the air stream; requires air density correction.
Critical Orifice or Device (Wedding, 1987)	Establishment of sonic flow in the orifice or device maintains a constant incoming flow despite downstream pressure changes.	Transfer standard; simple design provides a single flow rate setpoint.

Table 3-4 (continued)
Flow Measurement, Flow Control, and Flow Movers

	Device and Flow Range	Principle of Operation	Comments
	Laminar Flow Element (Bird et al., 1962; Lippmann, 1995)	Pressure drop across a precisely-machined array of channels is proportional to volumetric flow rate.	Transfer standard; requires fully-developed laminar flow which limits flow range; correction required if gas viscosity varies from calibration.
	Roots Meter (Lippmann, 1995)	Positive volume displacement is achieved by precisely-machined, tight-fitting opposed rotating lobes in a chamber.	Transfer or laboratory standard.
II.	***Flow Control***		
	Manual Flow Control (Rogers and Watson, 1989) (applicable from <10 to >100 L/min)	Manual adjustment of a valve between the filter and the vacuum pump.	Flow decreases as filter deposit accumulates.
	Mass Flow Control (Wedding, 1985) applicable from approx. 10 to >1200 L/min)	Sensing of heat transfer in moving air stream, which is proportional to velocity.	Air temperature changes cause volumetric flow to vary.
	Differential Pressure Volumetric Flow Control (applicable from 10 to 100 L/min) (Chow et al., 1993c)	Maintenance of constant pressure across a restriction.	Limited range of flows available with existing products.

Table 3-4 (continued)
Flow Measurement, Flow Control, and Flow Movers

	Device and Flow Range	Principle of Operation	Comments
	Critical Orifice or Critical Throat Volumetric Flow Control (no inherent restriction to flow range) (Wedding et al., 1987)	Choked (sonic) flow conditions in a restriction maintain constant flow. Critical throat requires lower pump capacities than critical orifice.	Air temperature changes cause small flow variations; very sensitive to upstream filter loads due to pressure drop across the filter changing the density of the air in the orifice throat but not affecting the velocity of sound (which remains constant).
	Constant Flow Controller (Lodge, 1995)	System uses a CFCASF filter (U.S. patent #5,317,930) as an orifice which feeds back the pressure drop across the filter flow controlling orifice.	Handles a large range of filter pressure drops with only a few percent change in flow. Permits the use of a wide variety of filters, in rotation, without recalibration.
	Constant Suction Unit (Schmidt and Wiltshire, 1955)	Open loop servo maintains constant flow through a pump having fixed displacement and operating at a constant speed.	Requires a vacuum regulator working in series with a positive-displacement air pump operating at a constant speed.
III.	***Generic Types of Air Movers***		
	Reciprocating Pump (few models operate at flows greater than 100 L/min. - Operation is constant-displacement) (Rubo and Furtado, 1989)	The rotary motion of the driveshaft is converted into reciprocating motion of a diaphragm or piston. The diaphragm or piston alternately forces air out of, and draws air into, a chamber equipped with one-way valves, to move it through a system.	Piston pumps generate higher outlet pressures and displace more of the air out of the chamber than diaphragm pumps. The flow pulsates unless special measures have been applied. Diaphragm failure modes include leaks or rupture.

Table 3-4 (continued)
Flow Measurement, Flow Control, and Flow Movers

Device and Flow Range	Principle of Operation	Comments
Rotary Vane Pump (wide range of flows available, from 1.0 L/min to greater than 1000 L/min; vane pumps operate as constant-displacement devices) (Rubo and Furtado, 1989)	Flexible or sliding vanes are rotated by an off-center shaft in a circular chamber, in an eccentric mode; air is compressed and forced out of the chamber as the vanes complete a rotation cycle.	Rotary vane pumps are subject to wear of their moving vane parts; the carbon-vane type exhausts carbon dust; main application is in vacuum mode.
Gear or Lobe Pump (wide range of flow rates available, from a few L/min to 100,000 L/min) (Rubo and Furtado, 1995)	Precisely-machined gears or lobes engage each other in a closely-fitting chamber; air is trapped between the engaging elements and forced out of the chamber.	Gear or lobe pumps are closely related to the Roots Meter type of flow measurement standard.
Radial or Axial Blower (flow range under minimal pressure gradient conditions is limited only by physical dimensions and rotation rate) (Rubo and Furtado, 1995)	The rotation of vanes or propellors transfers momentum to the air, moving it in tangential or axial directions.	Blowers rely on rotational velocity to maintain air motion through chambers which are not sealed against backward flow. Their ability to maintain pressure gradients is limited compared to other types of air movers.

Table 3-5
U.S. EPA Designated Reference and Equivalent Methods for PM_{10}

Reference/ Equivalent Method[a] (Designation No.)	Sampler Description	Federal Register Citation (Notice Date)
1. Reference method (RFPS-1087-062)	Wedding & Associates PM_{10} Critical Flow High-Volume Sampler (using a cyclone-type inlet, critical flow device, and 203 cm x 254 cm filters).	Vol. 52, 37366 (10/06/87)
2. Reference method (RFPS-1287-063)	Sierra-Andersen (SA) or General Metal Works (GMW) Model 1200 PM_{10} High-Volume Air Sampler System (using a SA- or GMW-1200 PM_{10} impaction-type size-selective inlet and 203 cm x 254 cm filters).	Vol. 52, 45684 (12/01/87) Vol. 53, 1062 (01/15/88)
3. Reference method (RFPS-1287-064)	Sierra-Andersen or General Metal Works Model 321-B PM_{10} High-Volume Air Sampler System (using a SA- or GMW-321-B PM_{10} impaction-type size-selective inlet and 203 cm x 254 cm filters).	Vol. 52, 45684 (12/01/87) Vol. 53, 1062 (01/15/88)
4. Reference method (RFPS-1287-065)	Sierra-Andersen or General Metal Works Model 321-C PM_{10} High-Volume Air Sampler System (using a SA- or GMW-321-C PM_{10} impaction-type size-selective inlet and 203 cm x 254 cm filters).	Vol. 52, 45684 (12/01/87) Vol. 53, 1062 (01/15/88)
5. Reference method (RFPS-0389-071)	Oregon DEQ Medium-Volume Sequential Filter Sampler for PM_{10} (using a SA-254 impaction-type PM_{10} inlet and 47 mm Teflon-membrane and quartz-fiber filters. Samples are collected simultaneously onto two filter substrates and can be programmed for up to six days of unattended operation and allow automatic filter-sequencing as filter overloading occurs).	Vol. 54, 12273 (03/24/89)

Table 3-5 (continued)
U.S. EPA Designated Reference and Equivalent Methods for PM_{10}

Reference/ Equivalent Method[a] (Designation No.)	Sampler Description	Federal Register Citation (Notice Date)
6. Reference method (RFPS-0389-073)	Sierra-Andersen Models SA-241 and SA-241M or General Metal Works Models G241 and GA-241M PM_{10} Low Volume Dichotomous Samplers (using a SA-246B or G246 impaction-type PM_{10} inlet, 2.5 µm virtual impactor assembly, and 37 mm $PM_{2.5}$ and coarse [PM_{10} minus $PM_{2.5}$] filter holders).	Vol. 54, 31247 (07/27/89)
7. Equivalent method (EQPM-0990-076)	Andersen Instruments Model FH621-N PM_{10} Beta Attenuation Monitor (using a SA-246B impaction-type PM_{10} inlet and 40 mm filter tape).	Vol. 55, 38387 (09/18/90)
8. Equivalent method (EQPM-1090-079)	Rupprecht & Patashnik TEOM Series 1400 and Series 1400a PM_{10} Monitor (using an impaction-type PM_{10} inlet, internal tapered element oscillating microbalance, and 12.7 mm diameter filter).	Vol. 55, 43406 (10/29/90)
9. Equivalent method (EQPM-0391-081)	Wedding & Associates PM_{10} Beta Gauge Automated Particle Sampler (using a cyclone-type PM_{10} inlet and 32 mm filter tape).	Vol. 56, 9216 (03/05/91)
10. Reference method (RFPS-0694-098)	Rupprecht & Patashnik Partisol Model 2000 Air Sampler (using an impaction-type PM_{10} inlet and 47 mm diameter filter).	Vol. 59, 35338 (07/11/94)

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[a] Code of Federal Regulations (1988).

Table 3-6
Filter-Based Particle Sampling Systems

Sampling System (Reference)	Particle Size (µm)	Inlet	Flow Rate (L/min.)	Sampling Surface	Filter Holders	Filter Media	Features
Western Region Air Quality Study (WRAQS) Sampler (Tombach et al., 1987)	PM_{15}	Aluminum high-volume impactor	113 out of 1,130	Aluminum and copper	Nuclepore polycarbonate in-line	47mm Teflon-membrane 47mm quartz-fiber	
	$PM_{2.5}$	Steel medium-volume cyclone)	113	Aluminum and copper	Nuclepore polycarbonate in-line	47mm Teflon-membrane 47mm quartz-fiber	
Size Classifying Isokinetic Sequential Aerosol (SCISAS) Sampler (Rogers et al., 1989)	PM_{15}	Aluminum high-volume impactor	113 out of 1,130	Aluminum and polyvinyl chloride	Nuclepore polycarbonate open-face	47mm Teflon-membrane 47mm quartz-fiber	Sequential sampling.
	$PM_{2.5}$	Steel medium-volume cyclone	113 out of 1,130	Stainless steel and aluminum	Nuclepore polycarbonate open-face	47mm Teflon-membrane 47mm quartz-fiber	

Table 3-6 (continued)
Filter-Based Particle Sampling Systems

Sampling System (Reference)	Particle Size (µm)	Inlet	Flow Rate (L/min.)	Sampling Surface	Filter Holders	Filter Media	Features
Southern California Air Quality Study (SCAQS) Sampler (Fitz and Zwicker, 1988; Fitz et al., 1989; Wolff et al., 1991)	PM_{10}	Aluminum medium-volume impactor	35 out of 113	Stainless steel and aluminum	Gelman stainless steel in-line	47mm Teflon-membrane 47mm quartz-fiber	Option to add 20 cm flow homogenizer.
	$PM_{2.5}$	Bendix 240 cyclone	35 out of 113	Teflon-coated aluminum	Gelman Stainless steel	47mm Teflon-membrane 47mm quartz-fiber 47mm impregnated quartz-fiber	Option to add 20 cm flow homogenizer.
				Teflon	Savillex PFA Teflon in-line	47mm nylon-membrane 47mm etched poly-carbonate	

Table 3-6 (continued)
Filter-Based Particle Sampling Systems

Sampling System (Reference)	Particle Size (μm)	Inlet	Flow Rate (L/min.)	Sampling Surface	Filter Holders	Filter Media	Features
Sequential Filter Sampler (SFS) (Chow et al., 1996a)	PM_{10}	Aluminum medium-volume impactor	20 out of 113	Aluminum	Nuclepore polycarbonate open-face	47mm Teflon-membrane 47mm quartz-fiber	Option to add nitric acid denuders in the sampling stream. Sequential sampling.
	$PM_{2.5}$	Aluminum medium-volume cyclone	20 out of 113	Teflon-coated aluminum	Nuclepore polycarbonate open-face	47mm Teflon-membrane 47mm quartz-fiber 47mm nylon-membrane 47mm impregnated cellulose-fiber	
California Acid Deposition Monitoring Program (CADMP) Dry Deposition Sampler (Chow et al., 1993c)	PM_{10}	Aluminum medium-volume impactor	20 out of 113	Aluminum	Savillex open-face	47mm Teflon-membrane 47mm impregnated cellulose-fiber	Includes nitric acid denuders. Sequential sampling.
	$PM_{2.5}$	Teflon-coated steel medium-volume cyclone	20 out of 113	PFA Teflon-coated aluminum	Savillex PFA Teflon open-face	47mm Teflon-membrane 47mm nylon-membrane	

Table 3-6 (continued)
Filter-Based Particle Sampling Systems

Sampling System (Reference)	Particle Size (µm)	Inlet	Flow Rate (L/min.)	Sampling Surface	Filter Holders	Filter Media	Features
Versatile Ambient Pollutant Sampler (VAPS) (Pinto et al., 1992, 1993; Mukerjee et al., 1993, 1994; Stevens et al., 1993a-b; Ma et al., 1994)	PM_{10}, $PM_{2.5}$	Teflon-coated aluminum low-volume elutriator and Teflon-coated aluminum low-volume virtual impactor	33	Teflon-coated aluminum	University Research Glassware glass filter pack (Model 2000-30F)	47mm Teflon-membrane 47mm etched polycarbonate membrane 47mm quartz-fiber	Includes annular denuders to capture nitric acid, nitrous acid, and sulfur dioxide; and polyurethane foam (PUF) to collect organic compounds.
California Institute of Technology Sampler (Solomon et al., 1988, 1989)	PM_{10}	Aluminum low-volume impactor	16.7	Stainless steel and aluminum	Gelman stainless steel in-line	47mm Teflon-membrane 47mm quartz-fiber	
	$PM_{2.5}$	Aluminum low-volume cyclone	22	Teflon-coated aluminum and glass	Gelman stainless steel in-line	47mm Teflon-membrane 47mm quartz-fiber 47mm nylon-membrane	
Stacked Filter Unit (SFU) (Cahill et al., 1990)	~$PM_{2.0}$ to $PM_{3.0}$	Large-pore etched polycarbonate filters	10	Polycarbonate	Nuclepore polycarbonate open-face	47mm etched poly-carbonate membrane 47mm Teflon-membrane	Uses large-pore etched polycarbonate filters as $PM_{2.5}$ inlet.

Table 3-6 (continued)
Filter-Based Particle Sampling Systems

Sampling System (Reference)	Particle Size (µm)	Inlet	Flow Rate (L/min.)	Sampling Surface	Filter Holders	Filter Media	Features
BYU Organic Sampling System (BOSS) (Eatough, 1993; Eatough et al., 1993a-b)	$PM_{2.5}$	Teflon-coated aluminum medium-volume cyclone	140 L/min through inlet and 35 L/min per channel	Teflon-coated stainless steel	University Research Glassware glass filter pack (Model 2000-30F)	47mm quartz-fiber 47mm activated-charcoal impregnated filter (CIF)	A multichannel diffusion denuder sampler to determine semi-volatile organic compounds.
BYU Big Organic Sampling System (BOSS) (Tang et al., 1995)	$PM_{2.5}$, $PM_{0.8}$, $PM_{0.4}$	Aluminum high-volume virtual impactor	1,130 L/min through inlet, with 11, 60, 93, and 200 L/min per channel	Teflon-coated stainless steel	University Research Glass filter pack (Model 2000-30F)	47mm quartz-fiber 47mm activated-charcoal impregnated filter (CIF) compounds	A multichannel diffusion denuder sampler to determine semi-volatile organic compounds.
Harvard/EPA Annular Denuder System (HEADS) (Koutrakis et al., 1989, 1991, 1992)	$PM_{2.5}$	Teflon-coated low-volume glass impactor	10	Glass	Graseby-Andersen open-face ring	37mm Teflon-membrane 37mm impregnated quartz-fiber	Includes sodium carbonate coated denuders to collect acidic gases (e.g., nitric acid, nitrous acid, sulfur dioxide, organic acids) and citric acid coated denuders to collect ammonia.

Table 3-6 (continued)
Filter-Based Particle Sampling Systems

Sampling System (Reference)	Particle Size (µm)	Inlet	Flow Rate (L/min.)	Sampling Surface	Filter Holders	Filter Media	Features
New York University Medical Center/ Sequential Acid Aerosol Sampling System (NYUMC/ SAASS) (Thurston et al., 1992)	$PM_{2.5}$	Teflon-coated glass low-volume impactor	4	Teflon-coated glass	Graseby-Andersen open-face rings	37mm Teflon-membrane 37mm nylon-membrane	Sequential sampling.
Minivol Portable Survey Sampler (Kemp, 1990; Chow and Watson, 1997b)	PM_{10}, $PM_{2.5}$	Nylon low-volume impactor	5	Polycarbonate	Nuclepore polycarbonate open-face	47mm Teflon-membrane 47mm quartz-fiber	Battery-powered sampler weighs 18 pounds.

Table 3-7
Test Specifications for $PM_{2.5}$ Equivalence to FRM[a]

Criteria	Specifications
Concentration Range	10 to 200 $\mu g/m^3$
Number of Test Sites	One for "Class I" monitors, two for "Class II" monitors
Number of Samplers	Three FRMs, three candidate samplers
Number of Samples	Class I 24-hour samples: R_j[b] $> 40\ \mu g/m^3$ and $R_j < 40\ \mu g/m^3$ Class I 48-hour samples: $R_j > 30\ \mu g/m^3$ and $R_j < 30\ \mu g/m^3$ Class II 24-hour samples: a. for $PM_{2.5}/PM_{10}$ ratio > 0.75: $R_j > 40\ \mu g/m^3$ and $R_j < 40\ \mu g/m^3$, b. for $PM_{2.5}/PM_{10}$ ratio < 0.40: $R_j > 30\ \mu g/m^3$ and $R_j < 30\ \mu g/m^3$, Class II 48-hour samples: a. for $PM_{2.5}/PM_{10}$ ratio > 0.75: $R_j > 30\ \mu g/m^3$ and $R_j < 30\ \mu g/m^3$, b. for $PM_{2.5}/PM_{10}$ ratio < 0.40: $R_j > 20\ \mu g/m^3$ and $R_j < 20\ \mu g/m^3$
Collocated Precision	2 $\mu g/m^3$ or 5% (largest)
Regression Slope	1 ± 0.05
Intercept	0 ± 1 $\mu g/m^3$
Correlation	0.97

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[a] U.S. EPA (1997b).

[b] R_j = the minimum number of acceptable sample sets per site for $PM_{2.5}$. R_j must be equal to or greater than 3.

Table 3-8
Continuous Aerosol Sampling and Analysis Systems

Continuous System	Quantity Measured	Methodology
I. Mass Monitors		
Beta Attenuation Monitor (BAM) (Lillienfeld and Dulchinos, 1972; Husar, 1974; Lillienfeld, 1975, 1976, 1979; Macias and Husar, 1976a-b; Jaklevic et al., 1981; Barnes et al., 1988; Wedding and Weigand, 1993; Williams et al., 1993)	Particle mass. Detection limit ~ 5 µg/m^3 for a one hour average	Beta rays (electrons with energies in the 0.01 to 0.1 MeV range) are attenuated according to an approximate exponential (Beer's Law) function of particulate mass, when they pass through deposits on a filter tape. Automated samplers utilize a continuous filter tape, first measuring the attenuation through the unexposed segment of tape to correct for blank attenuation. The tape is then exposed to ambient sample flow, accumulating a deposit. The beta attenuation measurement is repeated. The blank-corrected attenuation readings are converted to mass concentrations, with averaging times as short as 30 minutes.
Tapered Element Oscillating Microbalance (TEOM) (Patashnick and Rupprecht, 1990, 1991; Meyer et al., 1992)	Particle mass. Detection limit ~ 5 µg/m^3 for a five minute average.	Particles are continuously collected on a filter mounted on the tip of a glass element which oscillates in an applied electric field. The glass element is hollow, with the wider end fixed; air is drawn through the filter and through the element. The oscillation frequency of the glass element is maintained based on the feedback signal from an optical sensor. The resonant frequency of the element decreases as mass accumulates on the filter, directly measuring inertial mass. The typical signal averaging period is 5 minutes. Temperatures are maintained at a constant value, typically 30°C or 50°C, to minimize thermal expansion of the tapered element.

Table 3-8 (continued)
Continuous Aerosol Sampling and Analysis Systems

Continuous System	Quantity Measured	Methodology
Piezoelectric Microbalance (Olin and Sem, 1971; Wallace and Chuan, 1977; Fairchild and Wheat, 1984; Ward and Buttry, 1990; Williams et al., 1993)	Particle mass. Detection limit ~ 10 μg/m^3 for a one minute average.	Particles are deposited by inertial impaction or electrostatic precipitation onto the surface of a piezoelectric quartz crystal disk. The natural resonant frequency of the crystal decreases as particle mass accumulates. The changing frequency of the sampling crystal is electronically compared to a clean reference crystal, generating a signal that is proportional to the collected mass. The reference crystal also allows for temperature compensation.
II. Chemical-Specific Monitors		
Sulfur Analyzer, Chemiluminescent (Benner and Stedman, 1989, 1990; Schorran et al., 1994)	Sulfur dioxide and sulfate. Detection limit ~ 0.05 μg/m^3 for a 12 minute average	Sulfur species are converted to SO in a hydrogen flame; the SO is reacted with O_3 to produce an excited state of SO_2. Particulate- and gas-phase sulfur compounds are detected by chemiluminescence emission at 340 nm. Sulfur dioxide and sulfate can be selectively measured by applying a denuder difference approach. Temperature-controlled inlets can be used with chemiluminescent detectors in order to attribute the sulfur to particle-phase compounds based on their evaporation temperatures (e.g., H_2SO_4 at 120°C; NH_4HSO_4 and $(NH_4)_2SO_4$ at 300°C).

Table 3-8 (continued)
Continuous Aerosol Sampling and Analysis Systems

Continuous System	Quantity Measured	Methodology
Sulfur Analyzer, Flame Photometric Detection (FPD) (Cobourn et al., 1978; Huntzicker et al., 1978; Kittelson et al., 1978; Mueller and Collins, 1980; Tanner et al., 1980; Camp et al., 1982; Allen et al., 1984; Spengler et al., 1985; Appel et al., 1990)	Sulfur dioxide and sulfate. Detection limit ~ 1 µg/m^3 for a one hour average.	Sulfur species are combusted in a hydrogen flame, creating excited sulfur dimers (S_2*). Fluorescence emission near 400 nm is detected by a photomultiplier. The photomultiplier current is proportional to the concentration of sulfur in all species. With the quantitative addition of SF_6 to raise the response baseline, the signal/noise ratio can be increased by an order of magnitude. Temperature-controlled and denuder inlets are also used with FPD detectors in order to attribute the sulfur to particle-phase compounds based on their evaporation temperatures. Four out of five FPD systems agreed to within ± 5% in a one-week ambient sampling intercomparison.
Ammonia Analyzer, Fluorescence (Rapsomanikis et al., 1988; Genfa et al., 1989; Harrison and Msibi, 1994)	Gaseous ammonia. Detection limit < 1 µg/m^3 for a one hour average.	Sampled ammonia is removed from the airstream by a diffusion scrubber, dissolved in a buffered solution, and reacted with o-phtaldialdehyde and sulfite. The resulting i-sulfonatatoisoindole fluoresces when excited with 365 nm radiation, and the intensity of the 425 nm emission is monitored for quantification. The diffusion scrubber might be modified to pass particles while excluding ammonia gas to continuously quantify ammonium ions.
Nitric Acid Analyzer (Winer et al., 1974; Reid et al., 1980; Schiff et al., 1983; Anlauf et al., 1985, 1988; Burkhardt et al., 1988; Genfa et al., 1989; Kelly et al., 1990; McAdam et al., 1992; Solomon, 1994)	Gaseous nitric acid. Detection limit < 2 µg/m^3 for a one hour average.	Chemiluminescent, luminol, and tunable diode lasers detect nitrogen oxide, nitrogen dioxide, and nitric acid, respectively. Nitric acid can be reduced to NO or NO_2 prior to detection by the first two units. A sample stream denuded of nitric acid, nitrogen dioxide, and peroxyacetyl nitrate would leave only particulate nitrate. Heating these particles would create nitric acid for measurement by these detectors.

Table 3-8 (continued)
Continuous Aerosol Sampling and Analysis Systems

Continuous System	Quantity Measured	Methodology
In-Situ Thermal/Optical Carbon Analyzer Turpin et al., 1990; Turpin and Huntzicker, 1991)	Concentrations of organic and elemental carbon. Detection limit ~ 0.2 µg/m^3 for a two hour average.	This sampler provides on-line thermal/optical analysis of exposed quartz-fiber filters; the analysis principles are explained in Table 5-8. In the first step, organic carbon (OC) compounds are volatilized by heating the filter to 650°C in a helium atmosphere. The OC vapor-phase compounds are passed through a MnO_2 bed heated to 1000°C, where oxidation converts them to CO_2. The CO_2 is reduced to CH_4 in a nickel-firebrick methanator; the CH_4 is then measured in a flame ionization detector (FID). To quantify elemental carbon (EC), the temperature is then reduced to 350°C, and oxygen is added to the helium in order to oxidize the EC. The evolved CO_2 is reduced to CH_4 and measured by the same FID as applied to the OC. Light transmission through the filter is used to correct for charring (pyrolysis) of OC which may occur during the first analysis step. An identical quartz-fiber filter is exposed behind an absolute particle filter, allowing a correction for adsorbed OC vapor artifact. The measurement is calibrated by introducing CH_4 standards at the end of each cycle. The system is operated at 8.5 to 9.0 L/min and requires 80 to 240 minutes for a complete filter exposure and analysis cycle, depending on ambient concentrations.

Table 3-8 (continued)
Continuous Aerosol Sampling and Analysis Systems

Continuous System	Quantity Measured	Methodology
III. Other Aerosol Properties		
Integrating Nephelometer (Ahlquist and Charlson, 1967, 1969; Charlson et al., 1967, 1968, 1969, 1972, 1974a-b; Ensor and Waggoner, 1970; Charlson, 1972; Covert et al., 1972; Thielke et al., 1972; Rabinoff and Herman, 1973; Harrison, 1977, 1979; Waggoner and Charlson, 1977; Harrison and Mathai, 1981; Ruby and Waggoner, 1981; Larson et al., 1982; Hasan and Lewis, 1983; Waggoner et al., 1983; Hitzenberger et al., 1984; Rood et al., 1985, 1987, 1989; Ruby, 1985; Ruby et al., 1989; Horvath and Kaller, 1994)	*In-situ*, integrated light scattering from particles and gases; a direct estimate of the aerosol light-scattering coefficient, b_{scat}; lower detection limit ~ 1 mm^{-1} for a ten minute average.	Ambient gases and particles are continuously passed through an optical chamber; the chamber is generally in the form of a long cylinder illuminated from one side, perpendicular to the long axis of the chamber. The light source is located behind a lambertian diffuser and illuminates the aerosol at visible wavelengths. Light is scattered by particles in the chamber over angles ranging from 0° to 180°; mounted behind a series of baffles, a photomultiplier tube located at one end of the chamber detects and integrates the light scattered over about 9° to 171°. The light detected by the photomultiplier is usually limited by filters to wavelengths in the 500 to 600 nm range, corresponding to the response of the human eye. The instrument is calibrated by introducing gases of known index of refraction, which produce a known scattered energy flux. (For this purpose, halocarbon gases must now be replaced by non-ozone-reactive alternatives.) A typical signal averaging period is about 2 minutes.
Coefficient of Haze Sampler (COH) (ASTM, 1985; Herrick et al., 1989)	Optical density of particle deposited on a filter.	Particles are continuously deposited on a filter tape; a detector and light source are used to measure the blank-corrected optical density of the deposit. Though COH is reported in units of 1/1000 ft., the values are not traceable to primary standards.

Table 3-8 (continued)
Continuous Aerosol Sampling and Analysis Systems

Continuous System	Quantity Measured	Methodology
Aethalometer (Hansen et al., 1984, 1988, 1989; Hansen and Novakov, 1990a-b; Hansen and McMurry, 1990; Hansen and Rosen, 1990)	Light absorption, reported as concentration of elemental carbon. Detection limit ~ 10 ng/m^3 elemental carbon for a one minute average.	Ambient air is continuously passed through a quartz-fiber filter tape. A separate portion of the tape is not exposed to the sample stream, and provides an optical reference (blank). Light-absorbing particles such as black carbon cause attenuation of a light beam which is provided by a stabilized lamp behind a diffuser. The difference in attenuation between the exposed and blank segments of the filter tape is proportional to the amount of light-absorbing material collected on the tape. By assuming that all light-absorbing material is black carbon, and that the absorption coefficient of the black carbon is known and constant, the net attenuation signals can be converted into black carbon mass concentrations. The time resolution of the aethelometer is on the order of a fraction of a minute with a flow rate of 5 L/min.
Photoacoustic Spectroscopy (Terhune, 1977; Foot, 1979; Roessler and Faxvog, 1979; Truex and Anderson, 1979; Japar et al., 1984, 1989; Roessler, 1984; Adams, 1988; Adams et al., 1989a-b, 1990; Turpin et al., 1990; Moosmuller et al., 1994, 1995; Arnott et al., 1995)	Light absorption, reported as elemental carbon. Detection limit ~ 1.0 µg/m^3 for a one minute average.	Ambient air is aspirated through a resonant chamber, where it is illuminated by modulated (chopped) laser light at a visible wavelength (e.g., 514.5 nm). Light-absorbing particles, principally elemental carbon, absorb energy from the laser beam and transfer it as heating of the surrounding air. The expansion of the heated gas produces pressure pulses at the same frequency as the laser modulation. These pulses are detected by a microphone; its signal is proportional to the amount of absorbed energy. The illumination must be carefully chosen to avoid atmospheric gaseous absorption bands.

Table 3-8 (continued)
Continuous Aerosol Sampling and Analysis Systems

Continuous System	Quantity Measured	Methodology
Aerodynamic Particle Sizer	Number of particles in different size ranges.	Parallel laser beams measure the velocity lag of particles suspended in accelerating air flows.
Condensation Nuclei (CN) Counter (Pollak and Metnieks, 1959; Cheng, 1993)	Number of nucleating particles (particles larger than about 0.001 µm).	Particles are exposed to high supersaturations (150% or greater) of a working fluid such as water; droplets are subsequently nucleated, allowing detection of the particles by light scattering.
Differential Mobility Analyzer-Spectrometer (Yeh, 1993)	Number of nucleating particles in different size ranges (0.01 to 1.0 µm	Particles are classified according to their mobility in an electric field, which is a function of their size; a condensation nuclei counter then counts the population in a size “bin”.
Diffusion Battery (Fuchs, 1964; Cheng, 1993)	Number of nucleating particles in the sub-micrometer size range.	Particles are collected from laminar flows in tubes or channels according to their size-dependent Brownian diffusion mobilities. A condensation nuclei counter or other detector counts the transmitted particles. Data inversion gives input size distributions.

Table 3-8 (continued)
Continuous Aerosol Sampling and Analysis Systems

Continuous System	Quantity Measured	Methodology
Electrical Aerosol Analyzer (Whitby and Clark, 1966; Yeh,	Number of particles in the sub-micrometer size range (0.003 to 1.0 µm).	Particles are collected according to their size-dependent mobilities in an electric field. The collected particles are detected by their deposition of charge in an electrometer.
Optical Particle Counter/Size Spectrometer	Number of particles in the 0.1 to 50 µm size range.	Light scattered by individual particles traversing a light beam is detected at various angles; these signals are interpreted in terms of particle size via calibrations.
Time-of-Flight Mass Spectrometry (Nordmeyer and Prather, 1994; Prather et al., 1994)	Particle sizes and single particle compositions.	Particles in air are introduced into successively lower-pressure regions and acquire high velocities due to gas expansion. Particle size is evaluated by laser light scattering. The particles then enter a time-of-flight mass spectrometer.

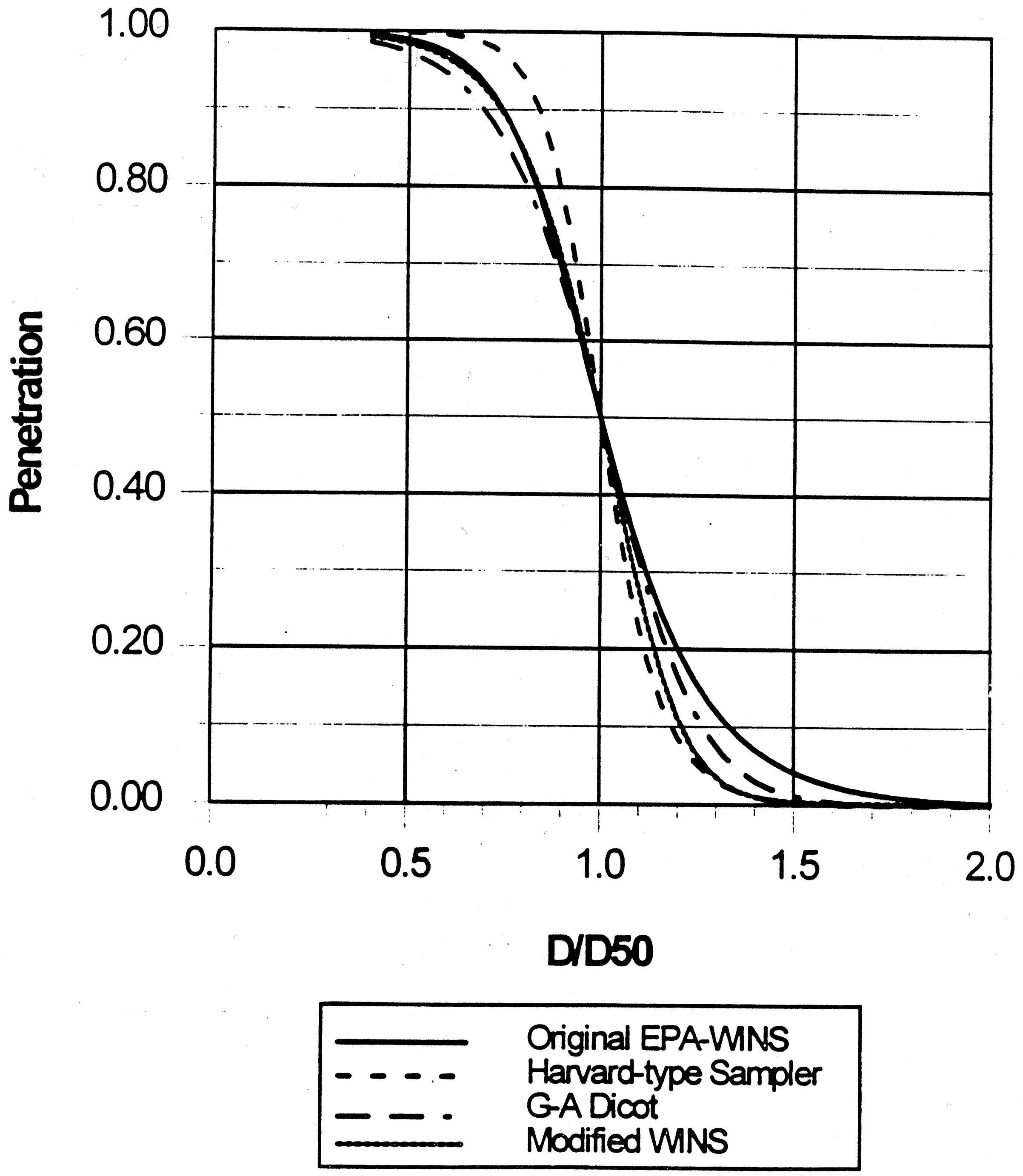


Figure 3-1. Characteristics of sampling effectiveness curves for WINS and other $PM_{2.5}$ inlets.

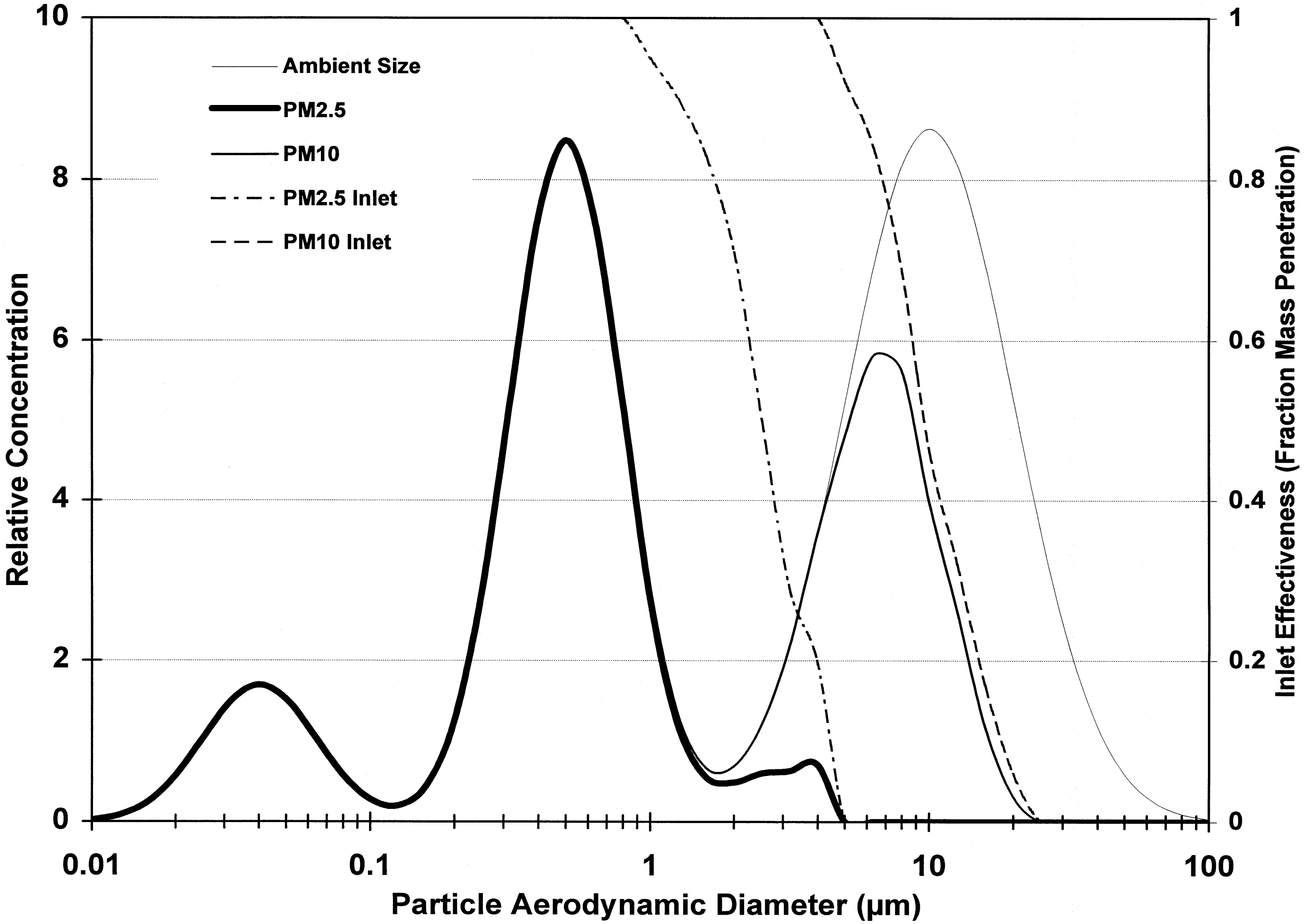


Figure 3-2. Changes in particle size distribution after passing through $PM_{2.5}$ and PM_{10} inlets.

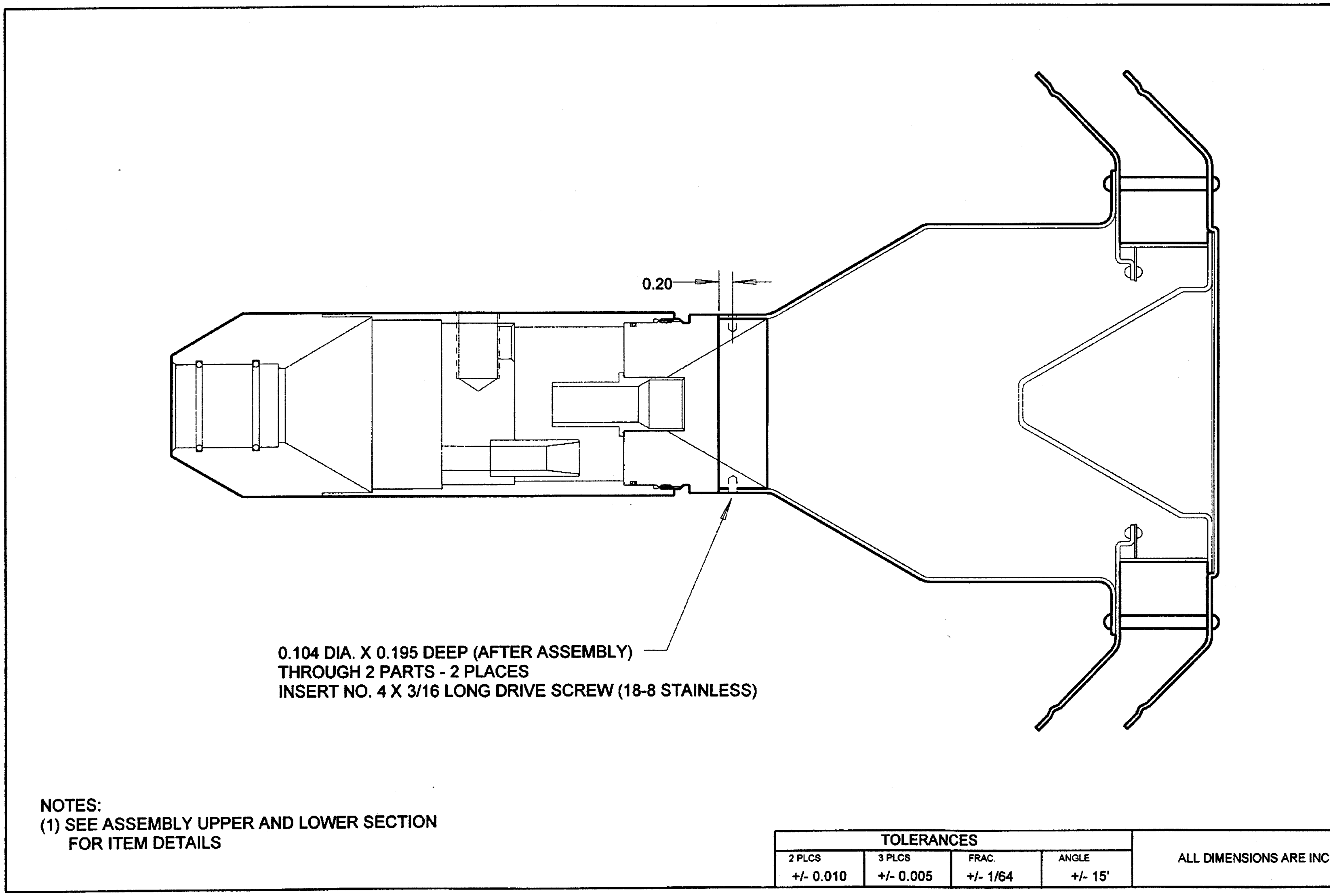
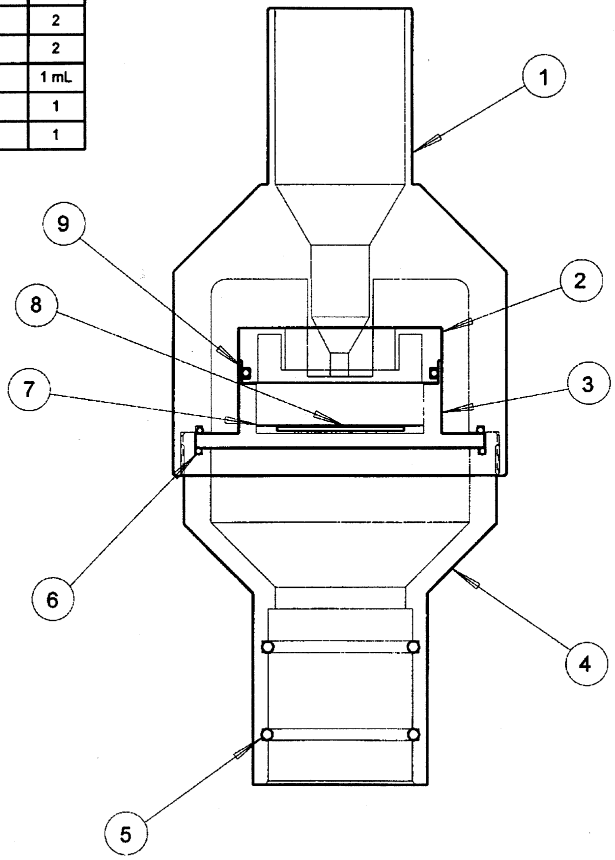


Figure 3-3. Schematic of a modified SA-246 PM$_{10}$ inlet.

ITEM	DESCRIPTION	QTY.
1	2.5-MICRON IMPACTOR HOUSING, UPPER (FIGURE L-21)	1
2	2.5-MICRON IMPACTOR WELL, UPPER (FIGURE L-22)	1
3	2.5-MICRON IMPACTOR WELL, LOWER (FIGURE L-23)	1
4	2.5-MICRON IMPACTOR HOUSING, LOWER (FIGURE L-24)	1
5	O-RING: AS568-026	2
6	O-RING: AS568-036 (VITON®)	2
7	IMPACTION OIL	1 mL
8	FILTER	1
9	O-RING: AS568-030 (VITON®)	1



VITON® IS A TRADEMARK OF DUPONT DOW ELASTOMERS L.L.C.

USE OF THIS NAME DOES NOT CONSTITUTE AN ENDORSEMENT OF EPA.

TOLERANCES			
2 PLCS	3 PLCS	FRAC.	ANGLE
+/- 0.010	+/- 0.005	+/- 1/64	+/- 15'

ALL DIMENSIONS ARE INCHES

Figure 3-4. Schematic of a WINS $PM_{2.5}$ inlet.

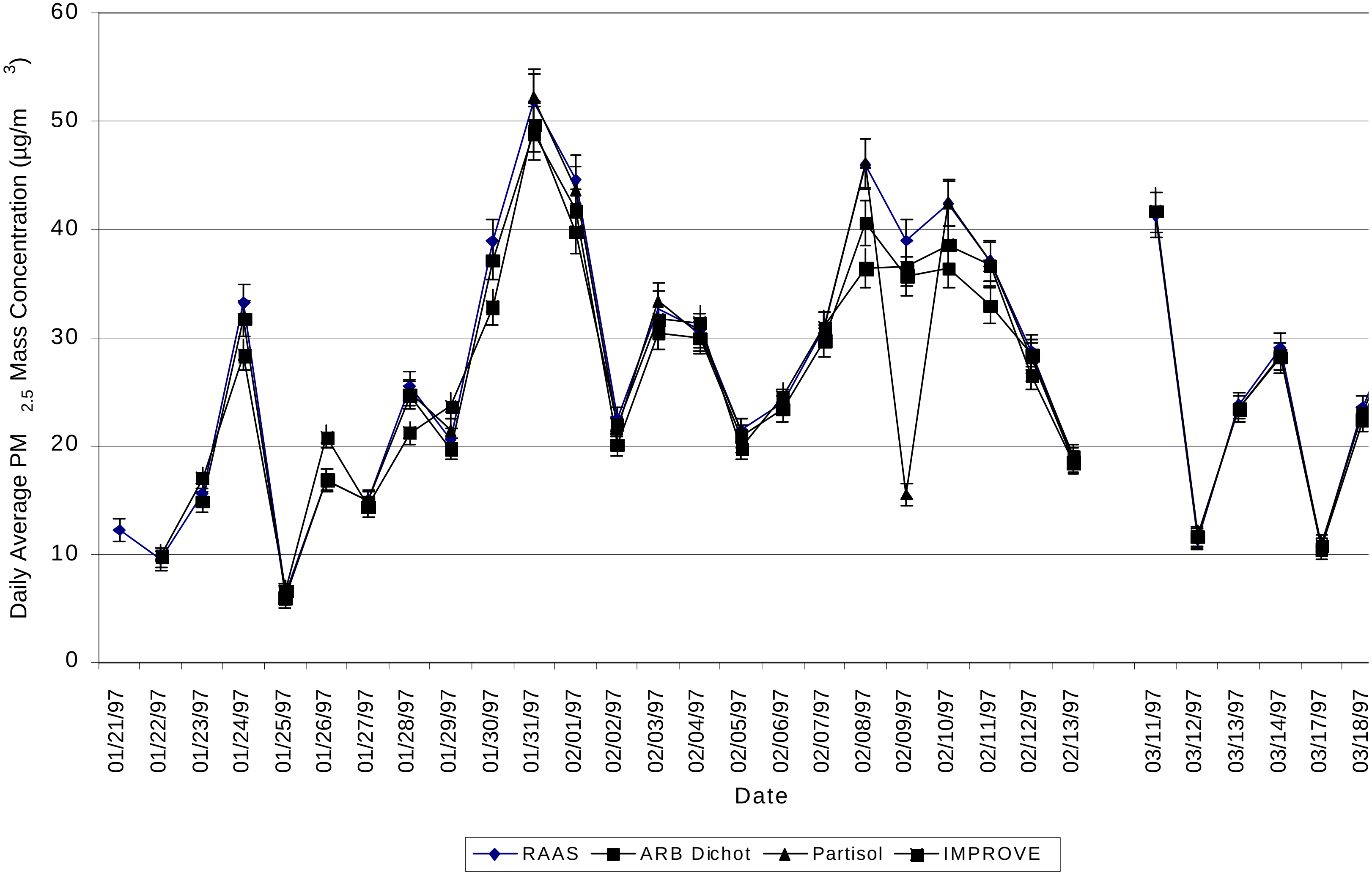


Figure 3-5. Comparison of $PM_{2.5}$ measurements from two WINS samplers with simultaneous measurements from dichotomous and IMPROVE samplers at the Bakersfield site between 01/21/97 and 03/19/97.

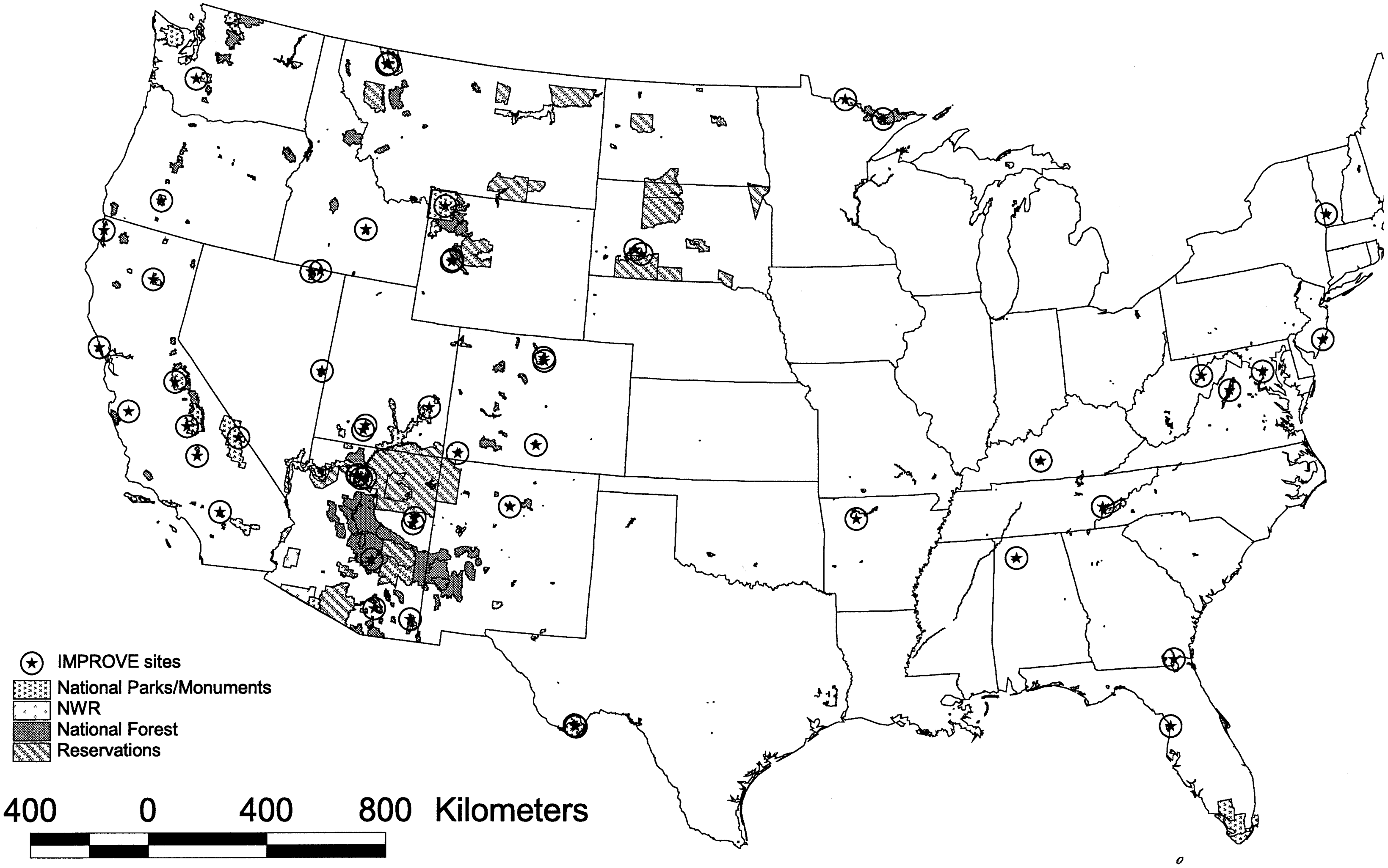


Figure 3-6. National parks and monuments, national wildlife refuges, national forests, Indian reservations, and IMPROVE background monitoring sites.

4.0 LABORATORY ANALYSIS METHODS

No matter how much air is drawn through a filter, and despite high particle loadings in the atmosphere, the amount of sample available for chemical analysis is small. The typical mass loadings on filters from low- to medium-volume samplers are less than 5 mg, and many of the chemical species of interest must be measured when less than 1 µg is present in the deposit.

Table 4-1 lists the minimum detectable limits for elemental, ionic, and carbon analysis methods that are commonly applied to aerosol filter samples. The values in Table 4-1 are nominal, and actual detection limits should be supplied by the laboratory performing the analysis *prior* to sampling. These are needed so that sample durations and flow rates can be adjusted to acquire sufficient sample for the intended analyses. These detection limits vary with sample duration, flow rate, and filter size. An example of detection limits for short-term (< 24-hour) sampling and recommended dilution volumes is shown Table 4-2.

Lodge (1989) and Appel (1993) provide extensive summaries of the principles, procedures, and results of these and other methods applied to the analysis of suspended particles. The following subsections define filter analysis protocols, specify filter handling and storage procedures, and describe chemical analysis methods. The most commonly applied aerosol analyses methods can be divided into the categories of mass, elements, ions, and carbon. For additional information to identify and quantify source/receptor relationships, organic compound, individual particle, and isotopic analyses methods can be applied.

4.1 Filter Analysis Protocols

The selection of appropriate analysis methods, filter media, and sampling hardware must be complemented with detailed sample handling and analysis procedures. Figure 4-1 illustrates a $PM_{2.5}$ sampling system that acquires gas and particle deposits on three different substrates sampled in parallel and in series. Figure 4-1 also shows the chemical analyses applied to different portions of each substrate. As shown, it is possible to obtain several different analyses on the same substrate, but it is not possible to obtain all desired chemical components from a single substrate.

Figure 4-2 summarizes the processes that might be applied in an aerosol characterization study involving multiple substrates and multiple samplers. Each box represents a set of actions that must be taken as part of the overall measurement process, and each box requires a detailed standard operating procedure. The extraction volumes given in Figure 4-2 are consistent with the MDLs listed in Table 4-1.

Depending on the study objectives and source mixture in the study area, different chemical species may need to be added to or omitted. Flow charts such as Figures 4-1 and 4-2 should be prepared prior to aerosol sampling for chemical analyses. They show precisely how samples are to be loaded, the extraction solutions needed, the recommended extraction volumes, and which analyses will be performed. This documentation reduces the possibility of submitting samples to the wrong analyses when filters are returned from the field and assists the data integration and data validation processes executed prior to data analysis and air quality modeling.

4.2 Filter Handling and Storage

No chemical analysis method, no matter how accurate or precise, can adequately represent atmospheric concentrations if the filters to which these methods are applied are improperly selected or handled. $PM_{2.5}$ or PM_{10} filter mass deposits are usually measured in micrograms (one-millionth of one gram). These are very small quantities, and even the slightest contamination can bias these mass measurements. Most chemical species that constitute $PM_{2.5}$ or PM_{10} are measured in nanograms (one-billionth of one gram). The risk of sample contamination when measuring these chemical components is 10 to 1,000 times greater than it is when measuring mass concentrations. Small biases in chemical concentrations can greatly affect the decisions that are made with respect to source apportionment or health effects, so extra precautions are warranted when selecting and using filters.

Teflon-membrane and quartz-fiber filters are most commonly used for the $PM_{2.5}$ and PM_{10} chemical analyses. Cellulose-fiber filters are easily impregnated with chemicals that absorb gaseous precursors, and etched polycarbonate-membrane filters are best suited for microscopic or individual particle analyses. Specific choices commonly applied in aerosol chemical characterization studies are: (1) Gelman (Ann Arbor, MI) polymethylpentane ringed, 2.0 µm pore size, 47 mm and 37 mm diameter polytetrafluorethylene (PTFE) Teflon-membrane filters (#R2PJ047, #R2PJ037) for mass by gravimetry, elements by x-ray fluorescence (XRF) or proton-induced x-ray emission (PIXE) spectroscopy, and optical absorption (b_{abs}) measurements by light absorption or filter transmission; (2) Gelman (Ann Arbor, MI) Nylasorb 47 mm diameter, nylon-membrane filters (#66509) for volatilized particle nitrate as well as total nitrate; (3) Pallflex (Putnam, CT) 47 mm diameter quartz-fiber filters (#2500 QAT-UP) for carbon by combustion methods as well as water-soluble chloride, nitrate, sulfate, ammonium, sodium, and potassium by ion chromatography (IC), automated colorimetry (AC), and atomic absorption spectrophotometry (AAS) measurements; and (4) Whatman 41 or Whatman 31ET (Hillsboro, OR) 47 mm diameter cellulose-fiber filters (#1441047) impregnated with adsorbing chemicals for sulfur dioxide, nitrogen dioxide, nitric acid, or ammonia measurements. These filters have been used primarily in the low-volume or medium-volume samplers described in Table 4-6. The manufacturer's identification numbers are important specifications since only these particular filters have been found to acceptably meet the requirements for chemical characterization in previous studies.

Most filters require treatment and representative chemical analyses (or "acceptance testing") when the deposits they acquire are intended for chemical speciation. Excessive blank levels and filter interferences discovered during or after several air quality studies have compromised the studies' results (Watson and Chow, 1994c). At least two filters from each lot (typically 100 filters) or a minimum of 2% of the filters purchased from the specified manufacturers should be analyzed for all species to verify that pre-established specifications have been met. Table 4-3 tabulates filter acceptance test results between 1992 and 1997 on over 1,000 lots of different filters. Average blank levels are typically less than 0.5 μg/filter for ions and less than 0.5 $\mu g/cm^2$ for carbon. Lots are rejected for chemical analysis when blank levels for individual species exceed 1 μg/filter. Table 4-3 shows that blank values are verified for various species on different filter types. Each filter should also be individually examined prior to labeling for discoloration, pinholes, creases, separation of ring, chaff or flashing, loose material, or other defects. For ringed Teflon filters, the diameter of the exposed area should be measured and should not deviate by more than ±1% of 40.538 mm.

Testing of sample media should continue throughout the course of a monitoring project. In addition to 2% to 5% of laboratory blanks, approximately 10% of all samples are designated as field blanks, and these follow all handling procedures except for actual sampling. The 46.2 ± 0.2 mm PTFE Teflon-membrane filter is part of the $PM_{2.5}$ FRM specification as set forth in 40 CFR part 50, Appendix L (U.S. EPA, 1997c). Table 4-4 summarizes the acceptance testing requirements for Teflon-membrane filters used in FRMs.

Teflon-membrane, quartz-fiber, and nylon-membrane filters often require pre-treatment prior to sampling, including:

- **Equilibrating Teflon-membrane filters:** On several occasions over the past 10 years (e.g., Tombach et al., 1987), batches of ringed Teflon-membrane filters have yielded variable (by up to 100 µg/filter over a few days) blank masses. As the time between manufacture and use increases, this variability decreases. Since filter manufacturers often minimize their long-term inventories of these filters and are manufacturing them on an as-ordered basis, this variability is being observed with greater frequency. A one-month storage period in a controlled environment, followed by one week of equilibration in the weighing environment (i.e., temperature within ±2 °C of 20 °C to 30 °C, relative humidity within ±5% of 30% to 40%), has been applied in several studies, and this appears to have reduced the variability to acceptable (within ±15 µg/filter for re-weights of 47 mm and 37 mm diameter filters) levels. Sets of Teflon-membrane filters which exceed two times the XRF detection limits for elements are rejected.

- **Pre-firing of quartz-fiber filters:** Quartz-fiber filters adsorb organic vapors over time. Blank quartz-fiber filters should be heated for at least three hours at 900 °C. A sample of each batch of 100 pre-fired filters is tested for carbon blank levels prior to sampling, and sets of filters with carbon levels exceeding 1 $\mu g/cm^2$ are re-fired or rejected. All pre-fired filters should be sealed and stored in a freezer prior to preparation for field sampling.

- **Washing nylon-membrane filters:** Nylon-membrane filters absorb nitric acid over time. Blank nylon-membrane filters should be soaked for four hours in 0.015 M sodium carbonate then rinsed in deionized distilled water (DDW) for 10 minutes, soaked overnight in DDW, rinsed three times in DDW, then dried in a vacuum oven at 60 °C for 5 to 10 minutes. Extraction efficiency tests have shown that the sodium carbonate IC eluent is needed to remove nitrates from the active sites of the nylon filter. Sets of washed nylon filters with nitrate levels exceeding 1 µg/filter should be rejected. Pre-washed nylon filters should be sealed and refrigerated prior to preparation for field sampling.

The results of all filter treatments, chemical analyses, and visual inspections should be recorded in a data base with the lot numbers. A set of filter IDs is assigned to each lot so that a record of acceptance testing can be associated with each sample.

In areas with large secondary inorganic aerosol contributions to $PM_{2.5,}$ precursor gases such as nitric acid (HNO_3), nitrogen dioxide (NO_2), ammonia (NH_3), or sulfur dioxide (SO_2) should also be monitored. Cellulose-fiber and quartz-fiber filters can be soaked in solutions of gas-adsorbing chemicals prior to sampling to collect these precursor gases. Several impregnation solutions have been used, and these solutions differ with respect to their reactive components and formulations. The criteria which must be met by the impregnation solution are: (1) availability of pure reagents; (2) stability of the impregnation solution composition before and after impregnation; (3) low degree of hazard or toxicity; (4) lack of interferences with other pollutants being sampled or with analytical methods; and (5) minimal effects of environmental factors such as temperature and water vapor content.

Sulfuric acid (Okita and Kanamori, 1971; Knapp et al., 1986), oxalic acid (Ferm, 1979; Ohira et al., 1976; Shendrikar and Lodge, 1975), phosphoric acid, sodium carbonate (Ferm, 1986), and citric acid (Stevens et al., 1985; Chow et al., 1993) have been used as the active agent in the sampling of ammonia on a variety of substrates. Citric acid impregnating solutions best meet the criteria described above.

Fung (1988) tested the ammonia absorption capacity of Whatman 41 cellulose-fiber filters impregnated with 0.13 µg of citric acid and 0.024 µg of glycerine. These filters adsorbed more than 4,000 µg of ammonia with better than 99% efficiency. Tests at temperatures ranging from –20 °C to 25 °C and at high and low relative humidities showed sampling efficiencies for ammonia in excess of 99%. Recent tests show that impregnated Whatman 31ET chromatographic paper absorbed more ammonia than Whatman 41 cellulose-fiber filters, making the Whatman 31ET better suited for sampling in ammonia-rich environments.

Potassium carbonate, sodium carbonate, or sodium chloride with glycerine have been used in impregnated filters for sulfur dioxide, nitric acid, or organic acid sampling (Forrest and Newman, 1973; Johnson and Atkins, 1975; Anlauf et al., 1985; Daum and Leahy, 1985; Hering et al., 1993; Tanner et al., 1993). The carbonate in the impregnating solution presents interferences to both the IC and AC analyses of extracts from these filters, however. In IC, the carbonate interferes with the nitrate peak and broadens the sulfate peak. In colorimetric methylthymol-blue analysis, the reaction of the MTB-Ba complex needs to be acidic and the carbonate raises the pH. Steps can be taken to alleviate these in the preparation of the filter extract prior to analysis.

Triethanolamine (TEA) has been used as an absorbent for nitrogen dioxide and to measure aerosol acidity (Dzubay et al., 1979). When used as a solution in a bubbler, TEA is a U.S. EPA equivalent method (No. EQN-1277-028) for monitoring nitrogen dioxide. Alary et al. (1974), Ohtsuka et al. (1978), Gotoh (1980), and Knapp et al. (1986) have applied TEA solutions to filter media such as Whatman 31ET chromatographic paper for the collection of nitrogen dioxide. The TEA is usually mixed with glycol or glycerine to improve its absorbing capacity (Doubrava and Blaha, 1980). Peroxyacetyl nitrate (PAN), organic nitrates, and sulfur dioxide are also collected by this substrate, and the nitrogen-containing compounds will appear as nitrate during analysis. TEA oxidizes in air and light, so impregnated filters must be stored in the dark in sealed containers.

Practical impregnation solutions consist of: (1) 25% citric acid and 5% glycerol (balance being water) for ammonia sampling; (2) 15% potassium carbonate and 5% glycerol solution (balance being water) for sulfur dioxide sampling; (3) 25% TEA and 5% ethylene glycol (balance being water) for nitrogen dioxide sampling; and (4) 5% sodium chloride (balance being water) for nitric acid sampling.

To impregnate filters, cellulose-fiber filter disks are immersed in the impregnating solution for approximately 30 minutes. These disks are then removed and placed in clean Petri slides for drying in a vacuum oven for five to ten minutes. One hundred of the dried impregnated filters are immediately sealed in polyethylene bags and placed under refrigeration for later loading into filter holders. One sample from each lot of citric acid filters is submitted to ammonium analysis prior to use. One sample from each lot should be extracted and analyzed prior to field sampling to assure that filter batches have not been contaminated. It is also useful to analyze each filter for a component of the impregnating solution (e.g., soluble potassium content on potassium carbonate impregnated filters) to verify that filters have acquired a sufficient amount of the adsorbing chemicals.

4.3 Mass Measurement Methods

Particulate mass concentration is the most commonly made measurement on aerosol samples. It is used to determine compliance with $PM_{2.5}$ and PM_{10} standards and to select certain samples for more detailed, and more expensive, chemical analyses. Gravimetric analysis is used almost exclusively to obtain mass measurements of filters in a laboratory environment. The guidance for weighing of 46.2 mm PTFE Teflon filters will be distributed as part of U.S. EPA's Quality Assurance Guidance Document.

Gravimetry measures the net mass on a filter by weighing the filter before and after sampling with a balance in a temperature- and relative humidity-controlled environment. To minimize particle volatilization and aerosol liquid water bias, $PM_{2.5}$ reference methods require that filters be equilibrated for 24 hours at a constant (within ±5%) relative humidity between 30% and 40% and at a constant (within ±2 °C) temperature between 20 °C and 23 °C, which is a more stringent requirement than for PM_{10} filter equilibration. PM_{10} filters are required to be equilibrated at 20% to 45% relative humidity (±5%) and 15 °C to 30 °C temperature (±3 °C). These filter equilibrium conditions are intended to minimize the liquid water associated with soluble compounds and to minimize the loss of volatile species. Nominal values of 30% RH and 20 °C best conserve the particle deposits during sample weighing. Accurate gravimetric analyses require the use of filters with low dielectric constants, high filter integrity, and inertness with respect to absorbing water vapor and other gases. Equilibration at low temperatures and relative humidities effectively removes liquid water associated with the particle deposit, but some particles may volatilize if they are exposed to ambient air for more than a day or two (Witz et al., 1988, 1990).

Gravimetric analysis of the filters needs to be performed with a microbalance (Feeney et al., 1984). The sensitivity and reliability of the electrobalance is about ±0.001 mg or ±1 μg, though tolerances on re-weights of Teflon-membrane filters are typically ±0.010 mg. These sensitive balances require isolation from vibration and air currents. Balances placed in laminar flow hoods with filtered air minimize contamination of filters from particles and gases in laboratory air. Ammonia produced by human breathing and cleaning solvents can neutralize acidic species that might have been captured on the filters.

The main interference in gravimetric analysis of filters results from electrostatic effects. Engelbrecht et al. (1980) found that residual charge on a filter could produce an electrostatic discharge between the filter on the pan and the metal casing of the electrobalance, which induces non-gravimetric forces. This charge can be removed from most filter media by exposing the filter to a low-level radioactive source (500 picocuries of polonium^{210}) prior to and during sample weighing.

Balance calibrations should be established before and after each weighing session using Class M and Class S standards traceable to National Institute of Standards and Technology (NIST, formerly National Bureau of Standards) mass standards, and they should be verified with a standard mass every ten filters. Approximately one out of ten filters should be re-weighed by a different person at a later time. These re-weights should be used to calculate the precision of the measurement as outlined by Watson et al. (1995a).

4.4 Elemental Analysis Methods

The most common interest in elemental composition derives from concerns about health effects and the utility of these elements to trace the sources of suspended particles. Instrumental neutron activation analysis (INAA), atomic absorption spectrophotometry (AAS), inductively coupled plasma with atomic emission spectroscopy (ICP-AES) or with mass spectroscopy (ICP-MS), photon-induced x-ray fluorescence (XRF), and proton induced x-ray emission (PIXE) have all been applied to elemental measurements of aerosol samples for atomic numbers ranging from 11 (sodium) to 92 (uranium). The subset of elemental MDLs listed in Table 4-1 includes those elements that have been detected in ambient air. AAS, ICP-AES, and ICP-MS are also appropriate for ionic measurements when the particles are extracted in DDW.

Since air filters contain very small particle deposits (20 to 100 μg/cm^2), preference is given to methods that can accommodate small sample sizes and that require little or no sample preparation or extensive operator time after the samples are loaded into the analyzer. XRF and PIXE leave the sample intact after analysis so that it can be submitted to additional examinations by other methods. To attain greatest efficiency and sensitivity, XRF and PIXE place the filters in a vacuum, and volatile compounds evaporate. Helium atmospheres are sometimes used to minimize, but not completely eliminate, particle volatilization.

In INAA (Dams et al., 1970; Zoller and Gordon, 1970; Olmez, 1989), a sample is irradiated in the core of a nuclear reactor for periods ranging from a few minutes to several hours. The neutron bombardment chemically transform many elements into radioactive isotopes. The energies of the gamma rays emitted by these isotopes identify them, and therefore their parent elements. The intensity of these gamma rays is proportional to the amount of the parent element present in the sample. Different irradiation times and cooling periods are used before counting with a germanium detector. INAA does not quantify some of the abundant species in ambient particulate matter such as silicon, nickel, tin, and lead. While INAA is technically nondestructive, sample preparation involves folding the sample tightly and sealing it in plastic, and the irradiation process makes the filter membrane brittle and radioactive. These factors limit the use of the sample for subsequent analyses.

In AAS (Ranweiler and Moyers, 1974; Fernandez, 1989), the sample is first extracted in a strong solvent to dissolve the solid material; the filter or a portion of it is also dissolved during this process. A few milliliters of this extract are introduced into a flame where the elements are vaporized. Most elements absorb light at certain wavelengths in the visible spectrum, and a light beam with wavelengths specific to the elements being measured is directed through the flame to be detected by a monochromater. The light absorbed by the flame containing the extract is compared with the absorption from known standards to quantify the elemental concentrations. AAS requires an individual analysis for each element, and a large filter or several filters are needed to obtain concentrations for all of the elements specified in Table 4-1. AAS is a useful complement to other methods, such as XRF and PIXE, for species such as beryllium, sodium, and magnesium which are not well-quantified by these methods. A typical double-beam AAS system is schematically illustrated in Figure 4-3. Airborne particles are chemically complex and do not dissolve easily into complete solution, regardless of the strength of the solvent. There is always a possibility that insoluble residues are left behind and soluble species may co-precipitate on them or on container walls.

In ICP-AES (Fassel and Kniseley, 1974; McQuaker et al., 1979; Lynch et al., 1980; Harman, 1989), the dissolved sample is introduced into an atmosphere of argon gas seeded with free electrons induced by high voltage from a surrounding Tesla coil. The high temperatures in the induced plasma raise valence electrons above their normally stable states. When these electrons return to their stable states, a photon of light is emitted which is unique to the element which was excited. This light is detected at specified wavelengths to identify the elements in the sample. ICP-AES acquires a large number of elemental concentrations using small sample volumes with acceptable detection limits for atmospheric samples. As with AAS, this method requires complete extraction and destruction of the sample.

Continued development of ICP-MS has resulted in increasing acceptance in environmental applications, especially for the determination of rare-earth elements in soils and sediments and trace elements from filter substrates (Tan and Horlick, 1986; Gray and Williams, 1987a-b). Ion species generated from ICP and from the sample matrix can produce a significant background at certain masses, resulting in formation of polyatomic ions that can limit the ability of ICP-MS to determine some elements of interest (Plantz, 1996). Cool plasma techniques have shown potential to detect elements at the ultra-trace level (Nham et al., 1996) and to minimize common molecular ion interferences (Sakata and Kawabata, 1994; Turner, 1994; Plantz, 1995).

As shown in Table 4-1, the detection limits of ICP-MS using a one-second scan are typically in the range of 10^{-3} ng/m^3, which is an order of magnitude lower than other elemental analysis methods. The instrument can also be set up to analyze a wide dynamic range of aerosol concentrations. Isotope analysis can also be performed with ICP-MS. Intercomparison studies need to be conducted to establish the comparability of ICP-MS with other non-destructive filter analysis methods.

In XRF (Dzubay and Stevens, 1975; Jaklevic et al., 1977) and PIXE (Cahill et al., 1990; Eldred, 1993), the filter deposit is irradiated by high energy x-rays (XRF) or protons (PIXE)which eject inner shell electrons from the atoms of each element in the sample. When a higher energy electron drops into the vacant lower energy orbital, a fluorescent x-ray photon is released. The energy of this photon is unique to each element, and the number of photons is proportional to the concentration of the element. Concentrations are quantified by comparing photon counts for a sample with those obtained from thin-film standards of known concentration.

Emitted x-rays with energies less than ~4 kev (affecting the elements sodium, magnesium, aluminum, silicon, phosphorus, sulfur, chlorine, and potassium) can be absorbed in the filter, in a thick particle deposit, or even by large particles in which these elements are contained. Very thick filters also scatter much of the excitation radiation or protons, thereby lowering the signal-to-noise ratio for XRF and PIXE. For this reason, thin membrane filters with deposits in the range of 10 to 50 μg/cm^2 provide the best accuracy and precision for XRF and PIXE analysis.

XRF methods can be broadly divided into two categories: wavelength dispersive (WDXRF), which utilizes crystal diffraction for observation of fluorescent x-rays, and energy dispersive (EDXRF), which uses a silicon semiconductor detector (Watson et al., 1997e). The WDXRF method is characterized by high spectral resolution, which minimizes peak overlaps. WDXRF requires high power excitation to overcome low sensitivity which results in excessive sample heating and potential degradation. Conversely, EDXRF features high sensitivity but less spectral resolution, requiring complex spectral deconvolution procedures.

XRF methods can be further categorized as direct/filtered excitation, where the x-ray beam from the tube is optionally filtered and then focused directly on the sample, or secondary target excitation, where the beam is focused on a target of material selected to produce x-rays of the desired energy. The secondary fluorescent radiation is then used to excite the samples. The direct/filtered approach has the advantage of delivering higher incident radiation flux to the sample for a given x-ray tube power, since about 99% of the incident energy is lost in a secondary fluorescor. The secondary fluorescor approach, however, produces a more nearly monochromatic excitation that reduces unwanted scatter from the filter, yielding better detection limits (Watson et al., 1997e).

XRF and PIXE are usually performed on Teflon-membrane filters for sodium, magnesium, aluminum, silicon, phosphorus, sulfur, chlorine, potassium, calcium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, arsenic, selenium, bromine, rubidium, strontium, yttrium, zirconium, molybdenum, palladium, silver, cadmium, indium, tin, antimony, barium, lanthanum, gold, mercury, thallium, lead, and uranium (as listed in Table 4-1).

An XRF system with secondary fluorescor is schematically illustrated in Figure 4-4. The x-ray output stability should be within ±0.25% for any 8-hour period within a 24-hour duration.

Analyses are typically controlled, spectra are acquired, and elemental concentrations are calculated by software on a computer which is interfaced to the analyzer. Separate XRF analyses are conducted on each sample to optimize detection limits for the specified elements. Figure 4-5 shows an example of an XRF spectrum.

Three types of XRF standards are used for calibration, performance testing, and auditing: (1) vacuum-deposited thin-film elements and compounds (Micromatter); (2) polymer films (Dzubay et al., 1981); and (3) NIST thin-glass films. The vacuum deposits cover the largest number of elements and are used to establish calibration curves. The polymer film and NIST standards are used as quality control measures. NIST produces the definitive standard reference material, but these are only available for the species aluminum, calcium, cobalt, copper, manganese, and silicon (SRM 1832), and iron, lead, potassium, silicon, titanium, and zinc (SRM 1833). A separate Micromatter thin-film standard is used to calibrate the system for each element.

Sensitivity factors (number of x-ray counts per $\mu g/cm^2$ of the element) are determined for each excitation condition. These factors are then adjusted for absorption of the incident and emitted radiation in the thin film. These sensitivity factors are plotted as a function of atomic number and a smooth curve is fitted to the experimental values. The calibration sensitivities are then read from these curves for the atomic numbers of each element in each excitation condition. Polymer film and NIST standards should be analyzed on a periodic basis using these sensitivity factors to verify both the standards and the stability of the instrument response. When deviations from specified values are greater than ±5%, the system should be re-calibrated.

The sensitivity factors are multiplied by the net peak intensities yielded by ambient samples to obtain the $\mu g/cm^2$ deposit for each element. The net peak intensity is obtained by: (1) subtracting background radiation; (2) subtracting spectral interferences; and (3) adjusting for x-ray absorption.

The elemental x-ray peaks reside on a background of radiation scattered from the sampling substrate. A model background is formed by averaging spectra obtained from several blank filters of the same type used in ambient sampling. It is important to retain blank filters for this purpose when XRF or PIXE analyses are anticipated. This model background has the same shape and features of the sample spectra (minus the elemental peaks) if the deposit mass is small relative to the substrate mass (Russ, 1977). This model background is normalized to an excitation radiation scatter peak in each sample spectrum to account for the difference in scatter intensity due to different masses.

The number and spacing of the characteristic x-ray lines relative to detector resolution are such that the peaks from one element can interfere with a peak from another element (Dzubay, 1986). A variety of methods has been used to subtract these peak overlaps (Arine et al., 1977; Parkes et al., 1979; Drane et al., 1983), including least squares fitting to library spectra, Gaussian and other mathematical functions, and the use of peak overlap coefficients.

Peak overlap coefficients are applied to aerosol deposits. The most important of these overlaps are the K-beta to K-alpha overlaps of elements which increase in atomic number from potassium to zirconium, the lead L-alpha to arsenic K-alpha interference, and the lead M line to sulfur K line interference. The ratios of overlap peaks to the primary peak are determined from the thin film standards for each element for the spectral regions of the remaining elements. These ratios are multiplied by the net peak intensity of the primary peak and subtracted from the spectral regions of other elements.

The ability of an x-ray to penetrate matter depends on the energy of the x-ray and the composition and thickness of the material. In general, lower energy x-rays, characteristic of light elements, are absorbed in matter to a much greater degree than higher energy x-rays. XRF analysis of air particulate samples has had widest application to samples collected on membrane-type filters such as Teflon- or polycarbonate-membrane filter substrates. These membrane filters collect the deposit on their surfaces, which eliminates biases due to absorption of x-rays by the filter material. These filters also have a low areal density which minimizes the scatter of incident x-rays, and their inherent trace element content is very low.

Quartz-fiber filters used for high-volume aerosol sampling do not exhibit these features. As noted earlier, blank elemental concentrations in quartz-fiber filters which have not undergone acceptance testing can be several orders of magnitude higher than the concentrations in the particulate deposits. They vary substantially among the different types of quartz-fiber filters available, and even within the same filter type and manufacturing lot. Blank impurity concentrations and their variabilities decrease the precision of background subtraction from the XRF spectral data, resulting in higher detection limits. Impurities observed in various types of glass- and quartz-fiber filters include aluminum, silicon, sulfur, chlorine, potassium, calcium, iron, nickel, copper, zinc, rubidium, strontium, molybdenum, barium, and lead. Concentrations for aluminum, silicon, and phosphorus cannot be determined for quartz-fiber filters because of the large silicon content of the filters.

Quartz-fiber filters also trap particles within the filter matrix, rather than on its surface. This causes absorption of x-rays within the filter fibers yielding lower concentrations than would otherwise be measured. The magnitude of this absorption increases exponentially as the atomic number of the analyte element decreases and varies from sample to sample. Absorption factors generally are 1.2 or less for iron and heavier elements, but can be from two to five for sulfur.

Quartz-fiber filters are much thicker than membrane filters resulting in the scattering of more x-rays with a consequent increase in background and degradation of detection limits. The increased x-ray scatter also overloads the x-ray detector which requires samples to be analyzed at a lowered x-ray intensity. These effects alone can result in degradation of detection limits by up to a factor of ten with respect to Teflon-membrane substrates.

Larger particles (>3 μm) collected during aerosol sampling have sufficient size to cause absorption of x-rays within the particles (Berry et al., 1969). Attenuation factors for $PM_{2.5}$ are generally negligible (Criss, 1976), even for the lightest elements, but these attenuations can be significant for coarse-fraction particles (particles with aerodynamic diameters from 2.5 to 10 µm). Methods have been developed to compensate for this absorption; these methods involve assumptions about particle size and composition that are accurate for most, but not all, cases of ambient air samples (Hunter and Rodes, 1972; Rodes and Hunter, 1972; Dzubay and Nelson, 1975).

During XRF or PIXE analysis, filters are removed from their Petri slides and placed with their deposit sides down into filter cassettes. These cassettes are loaded into a mechanism which exposes the filter deposits to protons for PIXE and x-rays for XRF. The sample chamber is evacuated and a computer program controls the positioning of the samples and the excitation conditions. The vacuum in the x-ray chamber and the heat induced by the absorption of x-rays can cause certain materials to volatilize. For this reason, labile species such as nitrate and organic carbon are better measured on a quartz-fiber filter that is sampled simultaneously with the Teflon-membrane filter.

Quality control standards and replicates from previous batches should be analyzed for every 10 to 20 samples. When quality control results differ from specifications by more than ±5%, or if the replicate concentrations differ from the original values (assuming they are at least 10 times detection limits) by more than ±10%, the samples should be re-analyzed.

XRF and PIXE are the most commonly used elemental analysis methods owing to their multi-element capabilities, relatively low cost, high detection limits, and preservation of the filter for other analyses. XRF sometimes needs to be supplemented with INAA when extremely low detection limits are needed, but the high cost of INAA prevents this method from being applied to large numbers of samples. Atomic absorption spectroscopy is a good alternative for water-soluble species, but it requires large dilution factors to measure many different elements. ICP is a viable alternative, but it is less desirable because of the expense required to extract the sample and the destruction of the filter sample.

4.5 Water-Soluble Ion Analysis Methods

Aerosol ions refer to chemical compounds which are soluble in water. The water-soluble portion of suspended particles associates itself with liquid water in the atmosphere when relative humidity increases, thereby changing the light scattering properties of these particles. Different emissions sources may also be distinguished by their soluble and non-soluble fractions, as in the case of soluble potassium. Gaseous precursors can also be converted to their ionic counterparts when they interact with chemicals impregnated on the filter material. Samples are generally extracted in DDW which is filtered to remove suspended particulate matter prior to analysis. Several simple ions, such as sodium, magnesium, potassium, and calcium can be quantified by AAS as described above. In practice, AAS has been very useful for measuring water-soluble potassium and sodium, which are important in apportioning sources of vegetative burning and sea salt, respectively. Polyatomic ions such as sulfate, nitrate, ammonium, and phosphate must be quantified by other methods such as ion chromatography (IC) and automated colorimetry (AC). Simple ions, such as chloride, and fluoride may also be measured by these methods along with the polyatomic ions. Some of these methods can be adapted to separately quantify metal ions with different valence states, such as iron (Fe II, Fe III), arsenic (As III, As V), and chromium (Cr III, Cr VI), that may have distinct effects on human health. When the aerosol deposit is suspected of being acidic, its hydrogen ion content can be determined by a pH electrode or by microtitration (Koutrakis et al., 1992). It is important to keep filter away from ammonia sources, such as human breath, to minimize neutralization of the acidic compounds.

All ion analysis methods require a fraction of the filter to be extracted in DDW and then filtered to remove insoluble residues prior to analysis. The extraction volume needs to be as small as possible, lest the solution become too dilute to detect the desired constituents at levels typical of those found in $PM_{2.5}$ or PM_{10}. Each square centimeter of filter should be extracted in no more than 2 ml of solvent for typical sampler flow rates of 16.7 L/min and sample durations of 24 hours. This often results in no more than 20 ml of extract which can be submitted to the different analytical methods, thereby giving preference to those methods which require only a small sample. Sufficient sample deposit must be acquired to account for the dilution volume required by each method.

The major sampling requirement for analysis of water-soluble species is that the filter material be hydrophilic, allowing the water to penetrate the filter and fully extract the desired chemical compounds. Small amounts of ethanol or other wetting agents are sometimes added to the filter surface to aid the wetting of hydrophobic filter materials, such as Teflon-membrane, but this introduces the potential for contamination of the sample. Zefluor filters have a porous Teflon backing which is difficult to distinguish from the membrane surface. Often, aerosol constituents are incorrectly sampled onto the back filter surface, where particles become trapped between the Teflon backing and the membrane surface, consequently hindering extraction efficiency. In this event, the membrane surface of each filter needs to be manually separated with forceps from the Teflon backing, and then both portions need to be extracted together.

When other analyses are to be performed on the same filter, the filter is first sectioned using a precision positioning jig attached to a paper cutter. Circular filters are usually cut in half for these analyses, so the results need to be multiplied by two to obtain the deposit on the entire filter. Filter materials for these analyses must be chosen so that they can be easily sectioned without damage to the filter or the deposit. The cutting blade should be cleaned between each filter cutting. The filter section is placed in an extraction vial which is capable of allowing it to be fully immersed in ~10 ml of solvent (the Falcon #2045 16 H 150 mm polystyrene vials are good choices). Each vial should be properly labeled with the sample ID and capped. Since much of the deposit is inside a fiber filter, agitation is needed to extract the water soluble particles into the solution. Experiments show that sonication for ~1 hour, shaking for ~1 hour, and aging under refrigeration for ~12 hours assures complete extraction of the deposited material in the solvent. The sonicator bath water needs to be periodically replaced or recirculated to prevent temperature increases from the dissipation of ultrasonic energy in the water. After extraction, these solutions should be stored under refrigeration prior to analysis. The unused filter sections should be placed back into their labeled containers, sealed airtight, and stored under refrigeration. These can be used for other analyses or they can serve as a backup if the original solution becomes contaminated or is insufficient for the planned ionic analyses. Figure 4-6 displays a flow diagram of filter extraction procedures.

The operating principle for AAS was described above. For potassium, the monochromater is set at 766.5 nm with a 2.0 nm bandpass. For sodium, the monochromater is set at 589.0 nm with a 0.7 nm bandpass. Approximately one to two ml of the extract are aspirated into an air/acetylene flame at approximately 0.5 mL/min. The output of the photomultiplier can be recorded on a data acquisition computer at rates of approximately two readings per second, and an overall 30 second average can be taken to attenuate variability due to flame fluctuation. This averaging should begin only after the sample has been aspirated for at least 30 seconds to assure that the flame has equilibrated. Two ml of DDW should be run between each sample to minimize carryover from the sample line. A blank and a known standard should be analyzed every ten samples to verify the span and baseline. Ten percent of the samples should be run in replicate at a later time, when there is sufficient extract, to evaluate analysis precision. American Chemical Society (ACS) reagent grade salts are dissolved in carefully measured volumes of DDW to create calibration and performance testing standards. Ionization interference is eliminated by addition of cesium chloride to samples and standard solutions.

IC can be used for both anions (fluoride, phosphate, chloride, nitrate, sulfate) and cations (potassium, ammonium, sodium) with separate columns (Chow and Watson, 1997c). Examples of filter impregnation and extraction or solutions applied in IC analysis are given in Table 4-5. Applied to aerosol samples, the anions are most commonly analyzed by IC with the cations being analyzed by a combination of AAS and AC. In IC (Small et al., 1975; Mulik et al., 1976, 1977; Butler et al., 1978; Mueller et al., 1978; Rich et al., 1978; Small, 1978), the sample extract passes through an ion-exchange column which separates the ions in time for individual quantification, usually by a electroconductivity detector. Figure 4-7 shows a schematic representation of the IC system. Prior to detection, the column effluent enters a suppressor column where the chemical composition of one element is altered, resulting in a matrix of low conductivity. The ions are identified by their elution/retention times and are quantified by the conductivity peak area or peak height. IC is especially desirable for particle samples because it provides results for several ions with a single analysis and it uses a small portion of the filter extract with low detection limits. Figure 4-8 shows an example of an IC anion chromatogram. IC analyses can be automated by interfacing to an automatic sampler which can conduct unattended analysis of as many as 400 samples (Tejada et al., 1978). Table 4-6 summarizes ion chromatographic analysis methods for water and air pollutants approved or recommended by U.S. EPA, the National Institute of Occupational Safety and Health (NIOSH), and the Occupational Safety and Health Adminstration (OSHA). These methods provide detailed procedures that can be applied to different types of environmental samples.

In IC, approximately 2 ml of the filter extract are injected into IC system. The resulting peak integrals are converted to concentrations using calibration curves derived from solution standards. Standard solutions of sodium chloride, sodium nitrate, and sodium sulfate can be prepared with reagent-grade salts which are dehydrated in a desiccator several hours prior to weighing. NIST-traceable simulated rain water standards (Standard Reference Materials: SRM 2694-I and SRM 2694-II) and the Environmental Research Associates (ERA) standard solution are available as independent quality control checks for the ions commonly measured by IC. Table 4-7 provides examples of quality assurance standards for commonly measured ionic species. Blanks and standards should be analyzed every ten samples, and one tenth of all $PM_{2.5}$ or PM_{10} extracts should be re-analyzed in the next analysis batch to estimate precision.

Depending on the dissociation of the species, the linear response range of the ion chromatographic system can be theoretically extended from 0.01 μg/mL to 100 μg/mL (Mulik and Sawicki, 1979). Strong acids or bases that are highly dissociated or ionized can be easily assayed by ion chromatography, whereas weak acids and bases that lack sufficient ionic character are more difficult to quantify. In practice, the linear response can only be assured within one to two orders of magnitude from the lowest calibration point. Overextending the concentration range will result in overestimation of the low concentrations or underestimation of the high concentrations. Calibration standards should span the entire range of sample concentrations, and separate calibration curves should be generated for samples with low and high concentrations.

Optimal calibration levels need to be established based on the typical concentration ranges in each area from which samples are acquired. For airborne particles, a six-point calibration curve is needed with concentrations of 0 (reagent blank), 0.05, 0.10, 0.20, 0.50, and 1.00 μg of analyte per mL of standard solution. Past experience shows that adequate linear response can be established within this concentration range. Over 80% of the airborne particle samples collected in urban and nonurban areas can be assayed within this concentration range without further dilution or with higher or lower standard concentrations.

A high-concentration calibration curve (e.g., 0, 1.0, 2.0, 5.0, 7.5, and 10.0 μg/mL) can be established for heavily-loaded ambient or source samples. Since the lowest point is 1.0 μg/mL instead of 0.05 μg/mL, this approach compromises the accuracy of low-concentration samples. It is advisable to reanalyze samples with concentrations lower than 1.0 μg/mL using low-concentration calibration curves (i.e., 0 to 1.0 μg/mL). For pristine environments with ultra-low ionic concentrations, standards of 0, 0.01, 0.02, 0.05, 0.10, and 0.50 μg/mL corresponding to the airborne particulate levels should be used to establish the calibration curve.

Standard stock solutions (typically 1,000 μg/mL) can be purchased or prepared with American Chemical Society (ACS) reagent-grade material. Solid reagent chemicals should be dried at 105 °C for one hour and cooled in a dessicator. Care must be taken when performing gravimetric analysis of these chemicals (to the nearest 0.1 mg) to minimize static charges and to prevent loss of solid chemicals after weighing. The standard stock solution should always be stored in the refrigerator and replaced at least annually for stable compounds such as sulfate, and more frequently for volatile compounds such as ammonium. Working standards of 10 and 100 μg/mL are used to prepare calibration standards on a monthly basis.

Calibration standards are prepared weekly or biweekly in routine operation of ionic analysis. New calibration standards need to be verified with the previous week's calibration standards to ensure consistency among the measurements. During instrument calibration, if any calibration point varies by more than ±5% of the specified value, the system should be rechecked or a new calibration standard should be prepared to ensure the accuracy of the analysis. Calibration data should be retained for reference and calibration curves should be generated for visual inspection. An example of a calibration curve is displayed in Figure 4-9.

Though automated data processing is usually applied to IC output, the chromatograms are too complex for such software to detect deviations from measurement assumptions. Each chromatogram should be examined individually to verify: (1) proper operational settings; (2) correct peak shapes and integration windows; (3) peak separation; (4) correct background subtraction; and (5) quality control sample comparisons. When values for replicates differ by more than ±10%, or values for standards differ by more than ±5%, all samples before and after these quality control checks should be re-analyzed. Individual samples with unusual peak shapes, background subtractions, or operating parameters should also be re-analyzed.

AC applies different colorimetric analyses to small samples volumes with automatic sample throughput. The most common ions measured are ammonium, chloride, nitrate, and sulfate (Butler et al., 1978; Mueller et al., 1978; Fung et al., 1979; Pyen and Fishman, 1979). Since IC provides multi-species analysis for the anions, ammonium is most commonly measured by AC.

The AC system is illustrated schematically in Figure 4-10. The heart of the automated colorimetric system is a peristaltic pump, which introduces air bubbles into the sample stream at known intervals. These bubbles separate samples in the continuous stream. Each sample is mixed with reagents and subjected to appropriate reaction periods before submission to a colorimeter. The ion being measured usually reacts to form a colored liquid. The liquid absorbance is related to the amount of the ion in the sample by Beer's Law. This absorbance is measured by a photomultiplier tube through an interference filter which is specific to the species being measured.

The standard AC technique can analyze ~50 samples per hour per channel, with minimal operator attention and relatively low maintenance and material costs. Several channels can be set up to simultaneously analyze several ions. The methylthymol-blue (MTB) method is applied to analyze sulfate. The reaction of sulfate with MTB-barium complex results in free ligand, which is measured colorimetrically at 460 nm. Nitrate is reduced to nitrite which reacts with sulfanilamide to form a diazo compound. This is then reacted to an azo dye for colorimetric determination at 520 nm. Ammonium is measured with the indophenol method. The sample is mixed sequentially with potassium sodium tartrate, sodium phenolate, sodium hypochlorite, sodium hydroxide, and sodium nitroprusside. The reaction results in a blue-colored solution with an absorbance measured at 630 nm.

Formaldehyde has been found to interfere with ammonium measurements when present in an amount which exceeds 20% of the ammonium content, and hydrogen sulfide interferes in concentrations which exceed 1 mg/mL. Nitrate and sulfate are also potential interferents when present at levels exceeding 100 times the ammonium concentration. These levels are rarely exceeded in ambient samples. The precipitation of hydroxides of heavy metals such as magnesium and calcium is prevented by the addition of disodium ethylenediamine-tetracetate (EDTA) to the sample stream (Chow et al., 1980; Chow, 1981). It was learned in the SUlfate Regional Experiment (SURE) (Mueller et al., 1983) that the auto-sampler should be enclosed in an atmosphere which is purged of ammonia by bubbling air through a phosphoric acid solution.

The automated colorimetric system requires a periodic standard calibration with the daily prepared reagents flowing through the system. Lower quantifiable limits of automatic colorimetry for sulfate and nitrate are an order of magnitude higher than those obtained with ion chromatography.

Intercomparison studies between automated colorimetry and ion chromatography have been conducted by Butler et al. (1978); Mueller et al. (1978); Fung et al. (1979); and Pyen and Fishman (1979). Butler et al. (1978) found excellent agreement between sulfate and nitrate measurements by automated colorimetry and ion chromatography. The accuracy of both methods is within the experimental errors, with higher blank values observed from automated colorimetric techniques. Comparable results were also obtained between the two methods by Fung et al. (1979). The choice between the two methods for sample analysis are dictated by sensitivity, scheduling, and cost constraints.

The major sampling requirement for analysis of water-soluble species is that the filter material be hydrophilic, allowing the water to penetrate the filter and fully extract the desired chemical compounds. Small amounts of ethanol or other wetting agents are sometimes added to the filter surface to aid the wetting of hydrophobic filter materials, but this introduces the potential for contamination of the sample.

4.6 Carbon Analysis Methods

Three classes of carbon are commonly measured in ambient aerosol samples collected on quartz-fiber filters: (1) organic, volatilized, or non-light absorbing carbon; (2) elemental or light-absorbing carbon; and (3) carbonate carbon. Carbonate carbon (i.e., K_2CO_3, Na_2CO_3, $MgCO_3$, $CaCO_3$) can be determined on a separate filter section by measurement of the carbon dioxide (CO_2) evolved upon acidification (Johnson et al., 1981). Though progress has been made in the quantification of specific organic chemical compounds in suspended particles (e.g., Rogge et al., 1991), sampling and analysis methods have not yet evolved for use in practical monitoring situations.

Several analytical methods for the separation of organic and elemental carbon in ambient and source particulate samples have been evaluated (Cadle and Groblicki, 1982; Stevens et al., 1982). These methods include: (1) solvent extraction of the organics followed by total carbon analysis (Gordon, 1974; Grosjean, 1975; Appel et al., 1976, 1979; Daisey et al., 1981; Muhlbaier and Williams, 1982; Japar et al., 1984); (2) nitric acid digestion of the organics followed by total carbon analysis (McCarthy and Moore, 1952; Kukreja and Bove, 1976; Pimenta and Wood, 1980); (3) absorption of radiation using an integrating plate to determine elemental carbon (variations of this method include infrared absorbance [Smith et al., 1975], Raman spectroscopy [Rosen et al., 1978], and visible absorbance [Lin et al., 1973; Weiss et al., 1979; Gerber, 1982; Heintzenberg, 1982]); (4) thermal combustion including both temperature-programmed (Muhlbaier and Williams, 1982) and step-wise pyrolysis followed by oxidation using either carbon dioxide or methane detection (Mueller et al., 1971, 1981; Patterson, 1973; Merz, 1978; Johnson and Huntzicker, 1979; Johnson et al., 1980; Malissa, 1979; Cadle et al., 1980a-b; Heisler et al., 1980a-b; Novakov, 1981; Tanner et al., 1982; Wolff et al., 1982); and (5) a combination of thermal and optical methods (Appel et al., 1976; Dod et al., 1979; Macias et al., 1979; Cadle et al., 1980a-b; Johnson et al., 1981; Novakov, 1982; Huntzicker et al., 1982; Rosen et al., 1982; Chow et al., 1993b).

Table 4-8 summarizes different carbon analysis methods along with their measurement principles. The definitions of organic and elemental carbon are operational (i.e., method dependent) and reflect the method and purpose of measurement (Grosjean, 1980). Elemental carbon is sometimes termed "soot", "graphitic carbon", or "black carbon." For studying visibility reduction, light-absorbing carbon is a more useful concept than elemental carbon. For source apportionment by receptor models, several consistent but distinct fractions of carbon in both source and receptor samples are desired, regardless of their light-absorbing or chemical properties. Differences in ratios of the carbon concentrations in these fractions form part of the source profile which distinguishes the contribution of one source from the contributions of other sources (Watson et al., 1994b).

Light-absorbing carbon is not entirely graphitic carbon, since there are many organic materials which absorb light (e.g., tar, motor oil, asphalt, coffee). Even the "graphitic" black carbon in the atmosphere has only a poorly developed graphitic structure with abundant surface chemical groups. "Elemental carbon" is a poor but common description of what is measured. For example, a substance of three-bond carbon molecules (e.g., pencil lead) is black and completely absorbs light, but four-bond carbon in a diamond is completely transparent and absorbs very little light. Both are pure, elemental carbon.

Chow et al. (1993b) document several variations of the thermal (T), thermal/optical reflectance (TOR), thermal/optical transmission (TOT), and thermal manganese oxidation (TMO) methods for organic and elemental carbon. The TOR, TOT, and TMO methods have been most commonly applied in aerosol studies for the analysis of organic and elemental carbon. Filter transmission analysis is often performed to estimate particle light absorption, which is proportional to the level of elemental carbon in the atmosphere. These methods are discussed in detail in the following subsections.

4.6.1 Thermal Manganese Oxidation Method for Carbon

The thermal manganese oxidation (TMO) method (Mueller et al., 1982; Fung, 1990) uses manganese dioxide present and in contact with the sample throughout the analysis, as the oxidizing agent. Temperature is relied upon to distinguish between organic and elemental carbon. Carbon evolving at 525 EC is classified as organic carbon, and carbon evolving at 850 EC is classified as elemental carbon.

This method has been used for the five year SCENES (the Subregional Cooperative Electric Utility, Department of Defense, National Park Services, and Environmental Protection Agency Study) (i.e., Mueller and McDade, 1986; Sutherland and Bhardwaja, 1986; Mueller et al., 1986; Watson et al., 1987) visibility network, as well as Southern California Air Quality Study (SCAQS, Chow et al., 1994a, 1994c-d; Watson et al., 1993, 1994b, 1994d).

4.1.2 Thermal Optical Reflectance/Transmission Method for Carbon

The thermal/optical reflectance (TOR) method of carbon analysis developed by Huntzicker et al. (1982) has been adapted by several laboratories for the quantification of organic and elemental carbon on quartz-fiber filter deposits. While the principle used by these laboratories is identical to that of Huntzicker et al. (1982), the details differ with respect to calibration standards, analysis time, temperature ramping, and volatilization/combustion temperatures.

In the most commonly applied version of the TOR method (Chow et al., 1993b), a filter is submitted to volatilization at temperatures ranging from ambient to 550 °C in a pure helium atmosphere, then to combustion at temperatures between 550 °C to 800 °C in a 2% oxygen and 98% helium atmosphere with several temperature ramping steps. The carbon which evolves at each temperature is converted to methane and quantified with a flame ionization detector. The reflectance from the deposit side of the filter punch is monitored throughout the analysis. This reflectance usually decreases during volatilization in the helium atmosphere owing to the pyrolysis of organic material. When oxygen is added, the reflectance increases as the light-absorbing carbon is combusted and removed. An example of the TOR thermogram is shown in Figure 4-11 (Chow et al., 1993b).

Organic carbon is defined as that which evolves prior to re-attainment of the original reflectance, and elemental carbon is defined as that which evolves after the original reflectance has been attained. By this definition, “organic carbon” is actually organic carbon that does not absorb light at the wavelength (632.8 nm) used and “elemental carbon” is light-absorbing carbon (Chow et al., 1993b). The thermal/optical transmission (TOT) method applies to the same thermal/optical carbon analysis method except that transmission instead of reflectance of the filter punch is measured.

Chow et al. (1993b) document several variations of the thermal (T), thermal/optical reflectance (TOR), thermal/optical transmission (TOT), and thermal manganese oxidation (TMO) methods for organic and elemental carbon. Chow et al. (1993b) also examine results from collocated elemental carbon measurements by optical absorption (OA), photoacoustic spectroscopy, and nonextractable mass. TOR was consistently higher than TMO for elemental carbon, especially in woodsmoke-dominated samples, where the disparity was as great as sevenfold. For the sum of organic and elemental carbon, these methods reported agreement within 5% to 15% for ambient and source samples (Houck et al., 1989; Kusko et al., 1989; Countess, 1990; Shah and Rau, 1991) and within 3% on carefully prepared standards. Evaluation of these methods then becomes a matter of assessing how they differentiate between organic and elemental carbon. The TMO method attributes more of the total carbon to organic carbon and less to elemental carbon than the TOR and TOT methods.

Comparisons among the results of the majority of these methods show that they yield comparable quantities of total carbon in aerosol samples, but the distinctions between organic and elemental carbon are quite different (Countess, 1990; Hering et al., 1990). None of them represents an ideal separation procedure of organic from elemental carbon.

4.1.3 Filter Transmission for Light Absorbing Carbon

Teflon-membrane and quartz-fiber filters can be submitted to a light transmission measurement before and after sampling on a transmission densitometer. An example of the measurement system is illustrated in Figure 4-12. Each filter is placed in a jig over a diffused vertical light beam. The spectral distribution is approximately Gaussian, peaking near 550 nm with full width at half maximum of about 150 nm. A detector is brought to a constant height above the filter and is precisely positioned with a shim to prevent contact with the filter itself. The filter density is displayed by the densitometer and can be later converted to transmittance. The same measurement is repeated on the exposed filter.

The instrument is calibrated with neutral density filters, and one of these standards is analyzed every 10 filters to verify instrument stability. If the response to these standards differs from specifications by more than 0.03 density units, the instrument is re-calibrated and the measurements are repeated on the previous ten samples. Replicate analyses are performed on one out of every ten samples, and when replicates deviate by more than ±0.05 density units from their original levels, samples are re-measured.

Informal intercomparisons among different filter transmission methods have shown high correlations of absorption, but differences of up to a factor of two in absolute values (Watson et al., 1988c). These differences are functions of: (1) the type of filter; (2) filter loading; (3) the chemical and physical nature of the deposit; (4) the wavelengths of light used; (5) calibration standards; and (6) light diffusing methods. At the current time, there is no agreement on which combination most accurately represents light absorption in the atmosphere. This method is applied with the knowledge that absolute differences in absorption may be found between the measurements made on Teflon-membrane and quartz-fiber filters and with respect to absolute absorption measurements made on the same samples in other laboratories.

With the limitations and precautions described above, laboratory analyses for the mass, elemental, ionic, and carbonaceous properties of suspended particles have matured to the point that they can be performed with commercially-available instruments, following established standard operating procedures, and with traceability to common standards. These analyses of trace substances still require extraordinary precautions to obtain accurate results.

4.7 Organic Speciation

Organic compounds are important components of particulate matter, whether in urban, rural, or remote areas. Most of the particulate organic carbon is believed to reside in the fine particle fraction. It has been reported (Gray et al., 1986) that in the Los Angeles area organic compounds constitute approximately 30% of the fine particle mass. Rogge et al. (1993a) analyzed $PM_{2.5}$ samples collected at four urban locations in southern California in 1982 to quantify individual organic compounds. Figure 4-13 shows the material balances that describe the chemical composition of ambient particulate matter for the most western (West Los Angeles) and most eastern (Rubidoux) sampling sites. During the summer photochemical smog season, the prevailing winds are from west to east. Under this meteorological condition, West Los Angeles is often upwind of the city, whereas Rubidoux is far downwind of the metropolitan area. Consequently, the concentrations of $PM_{2.5}$ mass and the secondary formation products such as nitrates and dicarboxylic acids are higher in Rubidoux than in West Los Angeles.

Rogge et al. (1993a) identified and quantified over 80 individual organic compounds in the $PM_{2.5}$ fraction, including n-alkanes, n-alkanoic acid, one n-alkenoic acid, one n-alkanal, aliphatic dicarboxylic acids, aromatic polycarboxylic acids, polycyclic aromatic hydrocarbons

(PAH), polycyclic aromatic ketones (PAK), polycyclic aromatic quinones (PAQ), diterpenoid acids, and some nitrogen-containing compounds. In general, the same type of organic compounds, although in different proportions, are found in direct emissions from various sources, such as diesel- and gasoline-powered vehicle exhaust, charbroilers and meat cooking operations, cigarette smoke, biogenic sources, etc. (Rogge, 1993; Rogge et al. 1991; 1993b-e). Thus, organic compounds are potentially valuable tracers for different emission sources, as well as for atmospheric transformation processes.

The collection of particulate organic matter can be accomplished using FRM instruments equipped with quartz-fiber or Teflon-impregnated glass fiber filters. However, since many organic compounds are distributed between the gas and particle phases, additional sampling techniques (such as a filter followed by solid adsorbents, for example, polyurethane foam (PUF), Tenax, or XAD resins) are required to measure both gaseous and particle phases of semi-volatile organic compounds (SVOC) (Zielinska and Fujita, 1994).

The most common method used for analysis of particulate matter collected on filters for speciated organic compounds is the extraction of a filter with a suitable organic solvent (or combination of solvents), followed by the analysis of the extract by gas chromatography (GC) combined with mass spectrometry (MS) or with other specific detectors. Combined GC/Fourier transform infrared (FTIR)/MS techniques or high performance liquid chromatography (HPLC)/MS techniques are also used.

Direct chemical analysis of the entire extractable fraction of particulate matter is not always possible because a large number of compounds of different polarity are present. The separation of particulate organic matter (POM) into various fractions according to chemical functionalities is a common preliminary step to chemical identification of individual compounds. Open-column liquid chromatography (LC) and liquid-liquid separation procedures have been the most widely used fractionation methods (Lee and Schuetzle, 1983). Open-column LC is very often followed by normal-phase HPLC, if the identification of less abundant components is required.

Much of the work on the identification of non-polar and semi-polar organics in airborne samples used bioassay-directed chemical analysis (Schuetzle and Lewtas, 1986), and focused on identification of fractions and compounds that are most likely to be significant in terms of human health. In particular, PAHs and their nitro-derivatives (nitroarenes) attracted considerable attention due to their mutagenic and, in some cases, carcinogenic properties. More than 100 PAHs have been identified in the $PM_{2.5}$ fraction of ambient particulate matter (Lee et al., 1981). While most of the nitroarenes found in ambient particles are also present in primary combustion-generated emissions, some are formed from their parent PAH in atmospheric nitration reactions (e.g., Arey et al., 1986; Ramdahl et al., 1986; Zielinska et al., 1989a-b).

Not much research has been done to chemically characterize the polar fraction in detail, even though polar material accounts for up to half the mass and mutagenicity of soluble ambient particulate organic matter (Atherholt et al., 1985; Gundel et al., 1993). The polar fraction of organic matter often remained analytically intractable because very few polar and labile species interact with conventional fractionation column packing materials and these species cannot be recovered quantitatively. New analytical techniques (e.g., HPLC/MS, MS/MS) or derivatization reactions need to be applied if the chemical constituents of polar particulate organic matter are to be identified and quantified.

Relatively little work has been done to characterize individual compounds or classes of compounds that might serve as tracers of specific sources of organic aerosol (e.g., Schauer et al., 1996; Rogge et al, 1993). In urban and rural atmospheres, as well as in the remote troposphere, organic composition corresponding to fingerprints of plant waxes, resin residues, and long-chain hydrocarbons from petroleum residues have been found (e.g., Gagosian et al., 1981; Simoneit, 1984; Mazurek et al., 1987, 1989, 1991; Simoneit et al., 1991; Rogge et al., 1993, 1993c, 1994). In addition, a variety of smaller, multi-functional compounds characteristic of gas-to-particle conversion have also been observed (e.g., Finlayson-Pitts and Pitts, 1986). These compounds tend to be present in the polar fraction of ambient organic aerosol particles, having been formed from atmospheric chemical reactions of less polar precursors. A summary of sampling and analysis methods for VOC and SVOC are summarized in Table 4-9 (Zielinska and Fujita, 1994). More research is needed to understand the chemical composition of polar organic fractions and to identify organic compounds that affect health.

4.8 Individual Particle Analysis

Single particles are characterized by optical or electron microscopy. Optical microscopy (Lee et al., 1979; Lee and Kelly, 1979, 1980; Janocko et al., 1982; Casuccio et al., 1983a-b, 1984, 1989; Dattner et al., 1983; Pettijohn et al., 1987; Lucass et al., 1988) is useful for coarse particles with sizes much larger than the wavelength of light (0.3 to 0.7 μm). Electron microscopy is needed to characterize particles in the $PM_{2.5}$ fraction for which particle size is comparable to visible light wavelengths. Computerized scanning and data acquisition methods are needed to characterize a number of particles sufficient to represent distributions on $PM_{2.5}$ samples.

Optical microscopes magnify coarse particles, consisting mostly of minerals, pollens, and metal fragments, so that they can be visually compared with known standards. Particle color, refractive index, birefringence, and crystallographic properties are also discernible by the use of wavelength-specific and polarizing filters on the illumination light. Chemical compositions are inferred from the similarity of observed particles to standard photographs that have also undergone laboratory analysis. To adequately determine these properties, each particle must be sufficiently isolated from other particles. This precludes the direct analysis of most aerosol filter samples.

Scanning Electron Microscopy (SEM) moves a focused electron beam under vacuum across a particle deposit (Kim and Hopke, 1988a-b). Vacuum is required because the electrons would otherwise be scattered off air molecules and make it difficult to get a sufficiently focused beam. The interaction of the electron beam with the sample produces various effects that can be monitored with suitable detectors. The resulting signals, which include secondary, backscattered, and Auger electrons, characteristic x-rays, as well as photo- and cathodoluminescence can be collected in synchronization with the position of the beam to provide highly detailed spatial and compositional information.

The secondary electron signal yields an image with a three-dimensional perspective, high depth-of-field, and the appearance of overhead illumination. The backscattered electron image is dependent on the number of backscattered electrons generated when the electron beam interacts with the sample. That is, higher-atomic-number elements generate more backscattered electrons (resulting in a brighter image) than low-atomic-number elements. Thus, the backscattered electron signal yields an image containing compositional information and offers the ability to discriminate between phases containing elements with different atomic numbers. An example of a secondary electron image and backscattered electron image of a particle is provided in Figure 4-14. The bright areas in the backscattered electron image are associated with a higher atomic number element.

Specific compositional information can be obtained through collection and processing of characteristic x-rays using energy dispersive spectroscopy (EDS) techniques (McCarthy, 1979). The EDS detector consists of a semiconducting crystal, usually lithium-drifted silicon, which is biased by means of gold electrodes placed on the front and rear surfaces. The electrical conductivity of the semiconducting crystal increases momentarily as an x-ray photon is absorbed and generates a charge avalanche. The magnitude of the corresponding electrical impulse in the semiconductor circuit is directly proportional to the energy of the x-ray photon. The individual pulses can be routed to different bins in a multi-channel analyzer or computer according to their magnitude. Thus, each time an x-ray strikes the detector, the electronic circuitry records the energy of the x-ray and counts its occurrence in a histogram display known as a spectrum. The energy and intensity (frequency of pulses) of the x-rays emitted by the sample can be recorded and the complete spectrum displayed in real time as it accumulates.

Owing to the high vacuum applied in SEM, windowless detectors can be used that detect light elements in addition to those elements typically characterized by XRF and PIXE including sodium, magnesium, aluminum, silicon, phosphorus, sulfur, chlorine, potassium, calcium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, arsenic, selenium, bromine, rubidium, strontium, yttrium, zirconium, molybdenum, palladium, silver, cadmium, indium, tin, antimony, barium, lanthanum, gold, mercury, thallium, lead, and uranium. Similar to XRF, peak overlap corrections must be applied to certain elements such as potassium/zirconium, lead/arsenic, lead/sulfur, zinc/sodium to properly identify peaks in the spectrum.

Figure 4-15 shows an example of the elemental information obtained using EDS techniques. Note that the elemental composition in Figure 4-15 is associated with a fly ash particle which is similar to that obtained from a soil particle.

By combining an SEM, an x-ray analyzer, and a digital scan generator under computer control, many individual particles can be automatically analyzed and categorized within a few hours (i.e., seconds per particle). Computer Controlled Scanning Electron Microscopy (CCSEM) allows for simultaneous measurement of individual particle size and elemental composition in an efficient manner (Schwoeble et al., 1988). With these attributes, CCSEM is capable of performing a quantitative microscopic analysis and grouping individual particles into particle classes based on their elemental composition, size, and shape. The CCSEM particle class data can then be summarized into number distribution and mass distribution tables.

4.8.1 CCSEM Analysis

CCSEM analysis requires that particles be deposited in a single layer onto a microscopically smooth substrate with a minimum of particles in contact. Ideally, these requirements would be fulfilled by collecting particles directly onto a polycarbonate-membrane filter while controlling the sampling time to achieve the ideal loading of 5 μg/cm^2. Alternatively, particles can be resuspended from a heavily loaded filter sample or from a bulk sample and re-deposited onto a polycarbonate-membrane filter using special sample preparation techniques. A schematic of a personal SEM is shown in Figure 4-16.

CCSEM analysis is typically performed using an accelerating voltage of 20 keV. The secondary electron or backscattered electron signal is used to create a viewing image and to determine when the electron beam is on a particle. The normal electron beam scanning motion is analogous to the motion of the electron beam in a conventional television set. With CCSEM, a digital scan generator converts the electron beam into a discrete stepping motion. Using computer control, the electron beam is “stepped” across the sample in an x, y pattern. At each point, the computer directs the electron beam to pause while the image intensity is compared to a threshold level. This comparison is used to determine whether the electron beam is “on” a particle (i.e., above a preset intensity threshold) or “off” a particle (i.e., below threshold), and to differentiate particles from background. When the signal is below the threshold level, the computer directs the digital scan generator to move the electron beam to a new (x, y) coordinate. This point-by-point approach permits the CCSEM analysis to be performed using “search” and “acquire” modes. A low grid-point density is used in the search mode, thereby increasing the area of the sample to be analyzed per unit time.

Once a coordinate is reached where the signal is above the threshold level (i.e., a particle is detected), “acquire” mode is enabled and the electron beam is driven across the particle in a preset pattern using a higher (more closely spaced) grid point density to determine the size of the particle. The measured average size is then compared to the acceptance criteria. If the particle is outside the size range of interest for a given magnification (i.e., too large or too small), the particle is rejected from the analysis. Otherwise, the average, maximum, and minimum diameters are recorded and collection of characteristic x-rays is initiated (Hooever et al., 1975; Kennedy and Lin, 1986, 1992).

Each particle’s physical diameter can be converted to an aerodynamic equivalent diameter using:

$$D_a(i) = \chi D_p(i) \tag{5-1}$$

for: $i = 1$ to n,

where: $D_a(i)$ = aerodynamic equivalent diameter in micrometers (μm) for particle i

$\chi\_$ = aerodynamic shape factor
$D_p(i)$ = average physical diameter (μm) for particle i
$Den(i)$ = particle density (pg/μm^3) for particle i
n = total number of particles detected during a CCSEM analysis.

The mass of an individual particle is calculated by multiplying the assigned density of the particle by its volume. The volume of the particle is calculated by assuming that it is oblate spheroid. Upon measurement of the particle size, the elemental composition of the particle is determined through collection of characteristic x-rays which are generated when the electron beam impinges on the particle. The energy and abundance of the x-rays emitted by the particle can be displayed as a spectrum of x-ray counts versus energy in real time. The elements present in the spectrum can be processed to obtain their relative concentrations. If an element is determined to be above background, the net x-ray counts for that element are determined by subtracting the background counts. Once the spectrum is processed and elements identified, a density is assigned to the particle based on its elemental composition (Johnson et al., 1987).

While most SEMs can easily resolve features in the nanometer (10^{-9} m) size range in the manual mode of operation, the practical effective lower limit in the automated mode is about 0.1 μm. With respect to elemental analysis, the detection limit under ideal conditions for an element in an individual feature is on the order of 0.5 wt.%. For a "rapid" CCSEM analysis (e.g., <5 second analysis) a detection limit between 1 and 5 wt.% is more appropriate. With respect to particle classes or types, the detection limit is based on the number of particles observed and the mode of occurrence. For example, if each particle has a homogeneous distribution of an element, say lead, at a low concentration (e.g., <1 wt. %), the CCSEM analysis may not be able to detect the lead. However, if the lead particles occur as discrete occurrences, then the CCSEM analysis has the ability to report concentrations down to the ppm level. It is even possible to report concentrations at lower concentrations using special analysis parameters.

A digital image of each particle can be acquired as part of the CCSEM analysis. Acquisition of the image is accomplished using the digital scan generator to control the electron beam raster and location (digital magnification and position), as well as the brightness and contrast of the image. Through the use of software, it is possible to dynamically alter the magnification and position of the digitally rastered beam to enable images of particles to be acquired during the analysis and stored on computer media (e.g., magnetic or optical disks) in an automated fashion (Stott and Chatfield, 1979; Henderson et al., 1989).

Automated stage control software can be used in conjunction with the CCSEM analysis. The software enables the stage to be moved to a new (random) location after particles in a field have been analyzed in an automated manner during the CCSEM analysis. In addition, the x and y coordinates for each particle analyzed by CCSEM are recorded and stored in the data base. This permits particles of specific interest (e.g., heavy element particles) to be re-examined in more detail using manual methods upon completion of the CCSEM analysis.

Quality Assurance (QA) for CCSEM consists of timely calibrations of the SEM (magnification and x-ray systems). The SEM magnification is calibrated using an NIST magnification standard or equivalent. The EDS x-ray analyzer is calibrated using element standards and following manufacture specifications or other accepted procedures. Particle measurement can be calibrated using particle standards such and NIST 10 μm spheres. Analysis of blank and replicate samples should be performed on a routine basis; and where appropriate, comparison of CCSEM results to other analytical data.

4.1.2 Electron Microprobes

Prior to the introduction of the SEM, electron microprobes had been developed to perform chemical analysis through use of an electron beam. The original electron microprobes were relatively simple instruments which focused a stationary electron beam onto the specimen. The analyst looked at the sample through microscope eyepieces and positioned the beam on the area to be analyzed. The x-rays produced were collected by wavelength dispersive spectrometers (WDS). Since these early microprobes did not produce images, they were not really microscopes at all. Rather, they were chemical analyzers which could analyze microscopic regions.

As SEMs became popular, electron microprobe manufactures began adding scanning capabilities to their instruments. Thus, modern SEMs and electron microprobes can both collect images and perform elemental analysis. A current-technology electron microprobe is similar to a high-sensitivity SEM that is equipped with multiple WDS units (Kennedy et al., 1996).

4.1.3 Transmission Electron Microscopy

Rather than scanning a focused beam of electrons on the surface of a sample like a SEM, a transmission electron microscope (TEM) shines a beam of electrons right through the sample and then, after magnification by a lens, projects the resulting image onto a phosphor screen. A TEM is analogous to a slide projector—it projects a magnified image of the specimen on the viewing screen. Since the electron beam must pass through the sample, it follows that the sample must be very thin and that the electron beam must be very energetic (typically 100 keV or higher).

Sample preparation for TEM analysis is more difficult than for SEM due to the complexity of TEM analysis. TEM analysis can provide much higher magnifications than a SEM. (Some TEMs can resolve individual atoms!) Also, TEMs have selected area electron diffraction (SAED) capabilities which offers the possibility to positively identify crystalline structures based on analysis of the SAED pattern. The TEM is ideally suited to characterize ultra-fine particles and has been used extensively over the past ten years in the analysis of asbestos fibers.

Table 4-1
Detection Limits of Air Filter Samples for Different Analytical Methods

	Minimum Detection Limit in ng/m^3 [a]									
Species	INAA [b,c]	XRF [b]	PIXE [b,d]	Flame AAS [e,f]	Graphite Furnace AAS [e,f]	ICP [e,g]	ICP/MS [b,h]	AC [e]	IC [e]	TOR
Ag	0.14	7	NA [i]	5	0.006	1	0.00432	NA	NA	NA
Al	29	6	14	36	0.01	24	0.04679	NA	NA	NA
As	0.2	1.0	1	120	0.2	60	0.03599	NA	NA	NA
Au	NA	2	NA	25	0.1	2.5	0.00468	NA	NA	NA
Ba	7	30	NA	10 [f]	0.05	0.06	0.00216	NA	NA	NA
Be	NA	NA	NA	2 [f]	0.06	0	0.00792	NA	NA	NA
Br	0.5	0.6	1	NA	NA	NA	0.16557	NA	NA	NA
Ca	113	2	5	1 [f]	0.06	0.05	0.03599	NA	NA	NA
Cd	5	7	NA	1	0.004	0.5	0.00396	NA	NA	NA
Ce	0.07	NA	NA	NA	NA	62	0.00108	NA	NA	NA
Cl	6	6	10	NA	NA	NA	NA	NA	NA	NA
Co	0.02	0.5	NA	7 [f]	0.02	1	0.00900	NA	NA	NA
Cr	0.2	1	2	2	0.01	2	0.09718	NA	NA	NA
Cs	0.04	NA	NA	NA	NA	NA	0.00180	NA	NA	NA
Cu	36	0.6	1	5	0.02	0.4	0.01080	NA	NA	NA
Eu	0.007	NA	NA	25	NA	0.10	0.00144	NA	NA	NA
Fe	5	0.8	2	5	0.02	0.6	0.01080	NA	NA	NA
Ga	0.6	1.1	1	62	NA	50	0.07558	NA	NA	NA
Hf	0.01	NA	NA	2,400	NA	19	NA	NA	NA	NA
Hg	NA	1	NA	600	25	31	0.12238	NA	NA	NA
I	1	NA	NA	NA	NA	NA	0.01908	NA	NA	NA
In	0.007	7	NA	37	NA	76	0.00504	NA	NA	NA
K	29	4	6	2 [f]	0.02	NA	0.01800	NA	NA	NA
La	0.06	36	NA	2,400	NA	12	0.00108	NA	NA	NA
Mg	360	NA	24	0.4	0.005	0.02	0.00360	NA	NA	NA
Mn	0.14	1.0	2	1	0.01	0.1	0.02843	NA	NA	NA
Mo	NA	1	6	37	0.02	6	0.01080	NA	NA	NA
Na	2	NA	72	0.2	< 0.06	NA	0.05399	NA	NA	NA
Ni	NA	0.5	1	6	0.1	2	0.08278	NA	NA	NA
P	NA	4	10	119,976	48	60	0.02160	NA	NA	NA
Pb	NA	1	4	12	0.06	12	0.02627	NA	NA	NA
Pd	NA	6	NA	12	NA	50	0.00576	NA	NA	NA
Rb	7	0.6	2	NA	NA	NA	0.00720	NA	NA	NA
S	7,199	2	10	NA	NA	12	0.05759	NA	NA	NA
Sb	0.07	11	NA	37	0	37	0.00612	NA	NA	NA

Table 4-1 (continued)
Detection Limits of Air Filter Samples for Different Analytical Methods

	Minimum Detection Limit in ng/m^3 [a]									
Species	INAA [b,c]	XRF [b]	PIXE [b,d]	Flame [e,f] AAS	Graphite Furnace [e,f] AAS	ICP [e,g]	ICP/MS [b,h]	AC [e]	IC [e]	TOR
Sc	0.001	NA	NA	60	NA	0.1	0.13677	NA	NA	NA
Se	0.07	0.7	1	120	0.6	30	0.99340	NA	NA	NA
Si	NA	4	11	102	0.1	4	0.02879	NA	NA	NA
Sm	0.01	NA	NA	2,400	NA	62	0.00540	NA	NA	NA
Sn	NA	10	NA	37	0.2	25	0.01512	NA	NA	NA
Sr	22	0.6	2	5	0.2	0.04	0.00288	NA	NA	NA
Ta	0.02	NA	NA	2,400	NA	31	NA	NA	NA	NA
Th	0.01	NA	NA	NA	NA	76	0.00216	NA	NA	NA
Ti	78	2	4	114	NA	0.4	0.03959	NA	NA	NA
Tl	NA	1	NA	25	0.1	50	0.00720	NA	NA	NA
U	NA	1	NA	29,994	NA	25	0.00180	NA	NA	NA
V	0.7	1	4	62	0.2	0.8	0.00432	NA	NA	NA
W	0.2	NA	NA	1,200	NA	37	NA	NA	NA	NA
Y	NA	0.7	NA	360	NA	0.1	0.00288	NA	NA	NA
Zn	4	0.6	1	1	0.001	1	0.06479	NA	NA	NA
Zr	NA	1.0	4	1,200	NA	0.7	0.00540	NA	NA	NA
Cl-	NA	NA	NA	NA	NA	NA	NA	NA	60	NA
NH4+	NA	NA	NA	NA	NA	NA	NA	60	NA	NA
NO3-	NA	NA	NA	NA	NA	NA	NA	NA	60	NA
SO4=	NA	NA	NA	NA	NA	NA	NA	NA	60	NA
Elemental Carbon	NA	NA	NA	NA	NA	NA	NA	NA	NA	120
Organic Carbon	NA	NA	NA	NA	NA	NA	NA	NA	NA	120

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[a] Minimum detection limit is three times the standard deviation of the blank for a filter of 1 mg/cm^2 areal density.
ICP-AES = Inductively Coupled Plasma Atomic Emission Spectroscopy.
ICP-MS = Inductively Coupled Plasma Mass Spectrometry.
AAS = Atomic Absorption Spectrophotometry.
PIXE = Proton Induced X-ray Emissions Analysis.
XRF = X-ray Fluorescence Analysis.
INAA = Instrumental Neutron Activation Analysis.
IC = Ion Chromatographic Analysis.
AC = Automated Colorimetric Analysis.
TOR = Thermal/Optical Reflectance Analysis.

[b] Concentration is based on 13.8 cm^2 deposit area for a 47 mm filter substrate, with a nominal flow rate of 16.67 L/min for 24-hour samples.

Table 4-1 (continued)
Detection Limits of Air Filter Samples for Different Analytical Methods

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[c] Olmez (1989).

[d] Cahill (1980).

[e] Concentration is based on the extraction of 1/2 of a 47mm filter in 15 ml of deionized-distilled water, with a nominal flow rate of 16.67 L/min for 24-hour samples.

[f] Fernandez (1989).

[g] Harman (1989).

[h] Tan and Horlick (1986), Jarvis et al. (1992).

[i] Chow et al. (1993b).

[j] Not available.

Table 4-2
Examples of Minimum Detection Limits for Low-Volume and Medium-Volume Gas and Particle Measurements

						Minimum Detection Limits (µg/m^3) as a Function of Flow Rate				
			Dilution/Area	Extraction	MDL	6-hour		12-hour		
Species	**Method**	**MDL/ Method**	**Factor**	**Vol (ml)**	**(µg/filter)**	**55 L/min**	**110 L/min**	**7 L/min**	**55 L/min**	**110 L/min**
Mass	Gravimetry	15 ug/filter	NA	NA	15	0.758	0.379	2.98	0.379	0.189
Chloride	IC	0.05 ug/ml	2	10.0	1.50	0.0505	0.0253	0.198	0.0253	0.0126
Nitrate	IC	0.05 ug/ml	2	10.0	1.50	0.0505	0.0253	0.198	0.0253	0.0126
Sulfate	IC	0.05 ug/ml	2	10.0	1.50	0.0505	0.0253	0.198	0.0253	0.0126
Ammonium	AC	0.05 ug/ml	2	10.0	1.50	0.0505	0.0253	0.198	0.0253	0.0126
Soluble Potassium	AA	0.07 ug/ml	2	10.0	2.10	0.071	0.0354	0.278	0.0354	0.0177
Nitric Acid (as NO_3^-)	IC	0.05 ug/ml	1	5.0	0.25	0.0126	0.0063	0.050	0.0063	0.0032
Ammonia (as NH_4^+)	AC	0.05 ug/ml	1	5.0	0.25	0.0126	0.0063	0.050	0.0063	0.0032
SO_2 (as $SO_4^=$)	IC	0.05 ug/ml	1	10.0	1.25	0.0253	0.0126	0.099	0.0126	0.0063
Total OC	TOR	0.82 ug/cm^2	13.8	NA	11.3	0.572	0.286	2.25	0.286	0.1429
Total EC	TOR	0.19 ug/cm^2	13.8	NA	2.62	0.132	0.0662	0.520	0.0662	0.0331
Al	XRF	0.0025 ug/cm^2	13.8	NA	0.138	0.0017	0.00087	0.007	0.0009	0.00044
Si	XRF	0.0014 ug/cm^2	13.8	NA	0.0869	0.0010	0.00049	0.004	0.0005	0.00024
P	XRF	0.0014 ug/cm^2	13.8	NA	0.0773	0.0010	0.00049	0.004	0.0005	0.00024
S	XRF	0.0012 ug/cm^2	13.8	NA	0.0690	0.00084	0.00042	0.003	0.0004	0.00021
Cl	XRF	0.0026 ug/cm^2	13.8	NA	0.138	0.0018	0.00091	0.007	0.0009	0.00045
K	XRF	0.0015 ug/cm^2	13.8	NA	0.0842	0.0010	0.00052	0.004	0.00052	0.00026
Ca	XRF	0.0011 ug/cm^2	13.8	NA	0.0621	0.00077	0.00038	0.003	0.00038	0.00019
Ti	XRF	0.00073 ug/cm^2	13.8	NA	0.0400	0.00051	0.00025	0.002	0.00025	0.00013
V	XRF	0.00062 ug/cm^2	13.8	NA	0.0345	0.00043	0.00022	0.002	0.00022	0.00011
Cr	XRF	0.00048 ug/cm^2	13.8	NA	0.0262	0.00033	0.00017	0.001	0.00017	0.000084
Mn	XRF	0.0004 ug/cm^2	13.8	NA	0.0221	0.00028	0.00014	0.001	0.00014	0.000070
Fe	XRF	0.00038 ug/cm^2	13.8	NA	0.0207	0.00026	0.00013	0.001	0.00013	0.000066
Co	XRF	0.00022 ug/cm^2	13.8	NA	0.0121	0.00015	0.00008	0.001	0.00008	0.000038
Ni	XRF	0.00022 ug/cm^2	13.8	NA	0.0123	0.00015	0.00008	0.001	0.00008	0.000038
Cu	XRF	0.00027 ug/cm^2	13.8	NA	0.0152	0.00019	0.00009	0.001	0.00009	0.000047

Table 4-2 (continued)
Examples of Minimum Detection Limits for Low-Volume and Medium-Volume Gas and Particle Measurements

						Minimum Detection Limits (µg/m^3) as a Function of Flow Rate				
			Dilution/Area	Extraction	MDL	6-hour		12-hour		
Species	Method	MDL/ Method	Factor	Vol (ml)	(µg/filter)	55 L/min	110 L/min	7 L/min	55 L/min	110 L/min
Zn	XRF	0.00027 ug/cm^2	13.8	NA	0.0152	0.0002	0.0001	0.001	0.0001	0.0000
Ga	XRF	0.00048 ug/cm^2	13.8	NA	0.0262	0.0003	0.0002	0.001	0.0002	0.0001
As	XRF	0.00039 ug/cm^2	13.8	NA	0.0221	0.0003	0.0001	0.001	0.0001	0.0001
Se	XRF	0.00031 ug/cm^2	13.8	NA	0.0166	0.0002	0.0001	0.001	0.0001	0.0001
Br	XRF	0.00025 ug/cm^2	13.8	NA	0.0138	0.0002	0.0001	0.001	0.0001	0.0000
Rb	XRF	0.00024 ug/cm^2	13.8	NA	0.0138	0.0002	0.0001	0.001	0.0001	0.0000
Sr	XRF	0.00028 ug/cm^2	13.8	NA	0.0152	0.0002	0.0001	0.001	0.0001	0.0000
Y	XRF	0.00033 ug/cm^2	13.8	NA	0.0179	0.0002	0.0001	0.001	0.0001	0.0001
Zr	XRF	0.00042 ug/cm^2	13.8	NA	0.0235	0.0003	0.0001	0.001	0.0001	0.0001
Mo	XRF	0.00067 ug/cm^2	13.8	NA	0.0373	0.0005	0.0002	0.002	0.0002	0.0001
Pd	XRF	0.0027 ug/cm^2	13.8	NA	0.1518	0.0019	0.0009	0.007	0.0009	0.0005
Ag	XRF	0.003 ug/cm^2	13.8	NA	0.1656	0.0021	0.0010	0.008	0.0010	0.0005
Cd	XRF	0.003 ug/cm^2	13.8	NA	0.1656	0.0021	0.0010	0.008	0.0010	0.0005
In	XRF	0.0034 ug/cm^2	13.8	NA	0.1794	0.0024	0.0012	0.009	0.0012	0.0006
Sn	XRF	0.0044 ug/cm^2	13.8	NA	0.2346	0.0031	0.0015	0.012	0.0015	0.0008
Sb	XRF	0.0045 ug/cm^2	13.8	NA	0.2484	0.0031	0.0016	0.012	0.0016	0.0008
Ba	XRF	0.0130 ug/cm^2	13.8	NA	0.7176	0.0091	0.0045	0.036	0.0045	0.0023
La	XRF	0.0160 ug/cm^2	13.8	NA	0.8556	0.0112	0.0056	0.044	0.0056	0.0028
Au	XRF	0.00077 ug/cm^2	13.8	NA	0.0428	0.0005	0.0003	0.002	0.0003	0.0001
Hg	XRF	0.00065 ug/cm^2	13.8	NA	0.0359	0.0005	0.0002	0.002	0.0002	0.0001
Tl	XRF	0.00062 ug/cm^2	13.8	NA	0.0345	0.0004	0.0002	0.002	0.0002	0.0001
Pb	XRF	0.00076 ug/cm^2	13.8	NA	0.0414	0.0005	0.0003	0.002	0.0003	0.0001
U	XRF	0.00059 ug/cm^2	13.8	NA	0.0317	0.0004	0.0002	0.002	0.0002	0.0001

Table 4-3
Summary of Filter Acceptance Test Results Performed at DRI's Environmental Analysis Facility between 1992 and 1997

Chemical Species	Filter	Manufacturer	Avg. ± Standard Deviation[a]	Unit	Filter Lots	Filters Tested
Chloride (Cl^-)	Teflon-membrane (2.0 μm pore size, 47 mm diameter, #R2PJ047)	Gelman Instrument Co. 600 S. Wagner Rd. Ann Arbor, MI 48106 (313) 665-0651	0.35 ± 0.27	μg/filter	84	112
	Quartz-fiber (#2500 QAT-UP)	Pallflex Production Co. Kennedy Dr. Putnam, CT 06260 (203) 928-7761	0.30 ± 0.23	μg/filter	638	1,276
Nitrate (NO_3^-)	Teflon-membrane (2.0 μm pore size, 47 mm diameter, #R2PJ047)	Gelman Instrument Co. 600 S. Wagner Rd. Ann Arbor, MI 48106 (313) 665-0651	0.032 ± 0.097	μg/filter	84	112
	Quartz-fiber (#2500 QAT-UP)	Pallflex Production Co. Kennedy Dr. Putnam, CT 06260 (203) 928-7761	0.12 ± 0.19	μg/filter	638	1,276
	Nylon-membrane (1.2 μm pore size, grade 66, 47 mm diameter, #00440)	Schleicher & Schuell, Inc. 543 Washington St. Keene, NH 03431 (800) 245-4029	0.21 ± 0.18	μg/filter	30	51

Table 4-3 (continued)
Summary of Filter Acceptance Test Results Performed at DRI's Environmental Analysis Facility between 1992 and 1997

Chemical Species	Filter	Manufacturer	Standard Avg. ± Deviation[a]	Unit	Filter Lots	Filters Tested
Nitrate (NO_3^-) (continued)	Whatman 31ET cellulose-fiber impregnated with NaCl (47 mm diameter)	Whatman, Inc. 9 Bridewell Place Clifton, NJ 07014 (201) 773-5800	0.37 ± 0.23	μg/filter	61	132
Sulfate ($SO_4^=$)	Teflon-membrane (2.0 μm pore size, 47 mm diameter, #R2PJ047)	Gelman Instrument Co. 600 S. Wagner Rd. Ann Arbor, MI 48106 (313) 665-0651	0.034 ± 0.087	μg/filter	84	112
	Quartz-fiber (#2500 QAT-UP)	Pallflex Production Co. Kennedy Dr. Putnam, CT 06260 (203) 928-7761	0.16 ± 0.21	μg/filter	650	1,300
	Nylon-membrane (1.2 μm pore size, grade 66, 47 mm diameter, #00440)	Schleicher & Schuell, Inc. 543 Washington St. Keene, NH 03431 (800) 245-4029	0.086 ± 0.13	μg/filter	30	51
	Whatman 41 cellulose-fiber impregnated with K_2CO_3 (47 mm diameter, #1441047)	Whatman, Inc. 9 Bridewell Place Clifton, NJ 07014 (201) 773-5800	0 ± 0[b]	μg/filter	64	32

Table 4-3 (continued)
Summary of Filter Acceptance Test Results Performed at DRI's Environmental Analysis Facility between 1992 and 1997

Chemical Species	Filter	Manufacturer	Standard Avg. ± Deviation[a]	Unit	Filter Lots	Filters Tested
Sulfate ($SO_4^=$) (continued)	Whatman 31ET cellulose-fiber impregnated with NaCl (47 mm diameter)	Whatman, Inc. 9 Bridewell Place Clifton, NJ 07014 (201) 773-5800	0 ± 0[c]	μg/filter	10	20
Ammonium (NH_4^+)	Teflon-membrane (2.0 μm pore size, 47 mm diameter, #R2PJ047)	Gelman Instrument Co. 600 S. Wagner Rd. Ann Arbor, MI 48106 (313) 665-0651	0.16 ± 0.18	μg/filter	86	121
	Quartz-fiber (#2500 QAT-UP)	Pallflex Production Co. Kennedy Dr. Putnam, CT 06260 (203) 928-7761	0.17 ± 0.20	μg/filter	650	1,300
	Whatman 41 cellulose-fiber impregnated with K_2CO_3 (47 mm diameter, #1441047)	Whatman, Inc. 9 Bridewell Place Clifton, NJ 07014 (201) 773-5800	0.09 ± 0.08[b]	μg/filter	8	10
	Whatman 31ET cellulose-fiber impregnated with NaCl (47 mm diameter)	Whatman, Inc. 9 Bridewell Place Clifton, NJ 07014 (201) 773-5800	0.40 ± 0.29[b]	μg/filter	55	110

Table 4-3 (continued)
Summary of Filter Acceptance Test Results Performed at DRI's Environmental Analysis Facility between 1992 and 1997

Chemical Species	Filter	Manufacturer	Standard Avg. ± Deviation[a]	Unit	Filter Lots	Filters Tested
Soluble Sodium (Na^+)	Teflon-membrane (2.0 μm pore size, 47 mm diameter, #R2PJ047)	Gelman Instrument Co. 600 S. Wagner Rd. Ann Arbor, MI 48106 (313) 665-0651	0.19 ± 0.19	μg/filter	86	114
	Quartz-fiber (#2500 QAT-UP)	Pallflex Production Co. Kennedy Dr. Putnam, CT 06260 (203) 928-7761	0.18 ± 0.24	μg/filter	656	1,312
Soluble Potassium (K^+)	Teflon-membrane (2.0 μm pore size, 47 mm diameter, #R2PJ047)	Gelman Instrument Co. 600 S. Wagner Rd. Ann Arbor, MI 48106 (313) 665-0651	0.15 ± 0.17	μg/filter	86	114
	Quartz-fiber (#2500 QAT-UP)	Pallflex Production Co. Kennedy Dr. Putnam, CT 06260 (203) 928-7761	0.05 ± 0.10	μg/filter	656	1,312
Organic Carbon (OC)	Quartz-fiber (#2500 QAT-UP)	Pallflex Production Co. Kennedy Dr. Putnam, CT 06260 (203) 928-7761	0.43 ± 0.30	$\mu g/cm^2$	1,111	2,223

Table 4-3 (continued)
Summary of Filter Acceptance Test Results Performed at DRI's Environmental Analysis Facility between 1992 and 1997

Chemical Species	Filter	Manufacturer	Avg. ± Standard Deviation[a]	Unit	Filter Lots	Filters Tested
Elemental Carbon (EC)	Quartz-fiber (#2500 QAT-UP)	Pallflex Production Co. Kennedy Dr. Putnam, CT 06260 (203) 928-7761	0.04 ± 0.15	μg/cm^2	1,111	2,223
Total Carbon (TC)	Quartz-fiber (#2500 QAT-UP)	Pallflex Production Co. Kennedy Dr. Putnam, CT 06260 (203) 928-7761	0.47 ± 0.37	μg/cm^2	1,111	2,223

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[a] The acceptance levels are: 1.0 μg/37mm or 47mm filter for anions and cations, 1.5 μg/cm^2 for organic carbon, 0.5 μg/cm^2 for elemental carbon, and 2.0 μg/cm^2 for total carbon.

[b] For the period of 1993 to 1995.

[c] For the period of 1996 to 1997.

Table 4-4
Federal Filter Specifications for $PM_{2.5}$ Sample Collection[a]

Attribute	Specification
Circular Size	46.2 ± 0.22 mm diameter.
Filter Medium	Polytetrafluoroethylene (PTFE Teflon) with integral support ring.
Support Ring	Polymethylpentene (PMP) or equivalent inert material. Outer diameter 46.2 ± 0.22 mm with ring thickness of 3.17 to 3.68 mm.
Pore Size	2 μm as measured by ASTM F316-94.
Filter Thickness	30 to 50 μm.
Maximum Pressure Drop	30 cm H_2O column with 16.67 L/min clean air flow on unexposed filter.
Collection Efficiency	Exceeding 99.7%, as measured by DOP test (ASTM D-2986-91) with 0.3 μm particles at the sampler's operating face velocity.
Filter Weight Stability	Drop test from 25 cm three times to verify loose surface particle contamination, which needs to be less than 20 μg for minimum of 0.1% per lot or 10 filters. Heat to 40 ± 20 °C for < 48 hours to test temperature stability, which needs to be less than 20 μg for minimum of 0.1% per lot or 10 filters.
Alkalinity	Less than 25 microequivalents per gram of filter.

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[a] U.S. EPA (1997c), 40 CFR part 50.

Table 4-5
Example of Filter Impregnation and Extraction Solutions Applied in Ion Chromatographic Analysis

Filter Medium	Impregnant Solution	Extraction Solution	Commonly Analyzed Ionic Species	Comments
Teflon-Membrane Filter	None	Distilled Deionized Water (DDW)	F^-, Cl^-, Br^-, NO_2^-, NO_3^-, $PO_4^=$, $SO_4^=$, Na^+, Mg^{++}, K^+, Ca^{++}, NH_4^+	Add 200μL of ethanol as wetting solution to filter surface prior to extraction.
Nylon-Membrane Filter	None	Sodium Carbonate / Sodium Bicarbonate	NO_3^-	
Quartz-Fiber Filter	None	Distilled Deionized Water	F^-, Cl^-, Br^-, NO_2^-, NO_3^-, $PO_4^=$, $SO_4^=$, Na^+, Mg^{++}, K^+, Ca^{++}, NH_4^+	
Impregnated Quartz-Fiber or Cellulose-Fiber Filter	Potassium Carbonate with 5% Glycerol	First Step: Add 0.1% Hydrogen Peroxide (H_2O_2) in 10 mL distilled deionized water Second Step: Perform 1 to 11 dilution on the 0.1% H_2O_2 extract	$SO_4^=$ as SO_2	Store 0.1% H_2O_2 solution for two days in the refrigerator to ensure complete SO_2 and SO_3^- oxidation.
Impregnated Quartz-Fiber or Cellulose-Fiber Filter	Citric Acid with 5% Glycerol	Distilled Deionized Water	NH_3 as NH_4^+	
Impregnated Quartz-Fiber or Cellulose-Fiber Filter	Sodium Chloride with 5% Glycerol	Sodium Carbonate / Sodium Bicarbonate	HNO_3 as NO_3^-	
Impregnated Quartz-Fiber or Cellulose-Fiber Filter	Triethanolamine (TEA) with 5% Glycerol	Sodium Carbonate / Sodium Bicarbonate	NO_2 as NO_2^-	

Table 4-6
Summary of Ion Chromatographic Compliance Testing Methods

Testing Methods	Matrix	Chemical Species	Concentration Range	References
U.S. EPA[a] Method 300A[b]	Water	Fluoride (F^-)	0.01[d] to 5.0 μg/mL	U.S. EPA (1991)
	Water	Chloride (Cl^-)	0.02[d] to 2.0 μg/mL	
	Water	Nitrite (NO_2^-)	0.004[d] to 10.0 μg/mL	
	Water	Bromide (Br^-)	0.01[d] to 5.0 μg/mL	
	Water	Nitrate (NO_3^-)	0.002[d] to 10.0 μg/mL	
	Water	Phosphate (PO_4^{-3})	0.003[d] to 5.0 μg/mL	
	Water	Sulfate ($SO_4^=$)	0.02[d] to 50.0 μg/mL	
U.S. EPA Method 300B[c]	Water	Chlorite (ClO_3^-)	0.01[d] to 5 μg/ml	U.S. EPA (1991)
	Water	Chlorate (ClO_2^-)	0.02[d] to 5 μg/ml	Pfaff et al. (1991)
	Water	Bromate (BrO_3^-)	0.03[d] to 5 μg/ml	
U.S. EPA Method 300.7[b]	Water	Sodium (Na^{++})	0.03[d] to 1.00	U.S. EPA (1985, 1986a, 1986b)
	Water	Magnesium (Mg^{++})	0.02[d] to 1.00	
	Water	Potassium (K^+)	0.01[d] to 1.00	Bachman et al. (1986)
	Water	Calcium (Ca^{++})	0.02[d] to 3.00	
	Water	Ammonium (NH_4^+)	0.03[d] to 2.00	
U.S. EPA Method 218.6 and U.S. EPA Method 1636	Water	Hexavalent Chromine (Cr IV) determined as $CrO_4^=$	0.001 to 5 μg/mL	U.S. EPA (1994, 1995)

Table 4-6 (continued)
Summary of Ion Chromatographic Compliance Testing Methods

Testing Methods	Matrix	Chemical Species	Concentration Range	References
NIOSH[e] 6004	Air	Sulfur Dioxide (SO_2) determined as sulfate ($SO_4^{=}$) sulfite ($CrO_4^{=}$)	1.1 to 20 μg/mL	NIOSH Manual of Analytical Methods (1994a)
NIOSH 6011	Air	Chlorine (Cl) determined as chloride (Cl^-)	0.06 to 1.5 μg/mL	NIOSH Manual of Analytical Methods (1994b)
		Bromine (Br) determined as bromide (Br^-)	0.06 to 1.5 μg/mL	
NIOSH Method 7903	Air	Inorganic Acids determined as common anions:		NIOSH Manual of Analytical Methods (1994c)
		Hydrofluoric Acid (HF) as F^-,	0.03 to 13.3 μg/mL	
		Hydrochloric Acid (HCl) as Al^-,	0.03 to 13.3 μg/mL	
		Phosphoric Acid (H_3PO_4) as $PO_4^{=}$,	0.2 to 6.7 μg/mL	
		Hydrobromic Acid (HBr) as Br^-,	0.2 to 64 μg/mL	
		Nitric Acid (HNO_3) as NO_3^-,	0.2 to 33.3 μg/mL	
		Sulfuric Acid ($H_2SO_4^{=}$) as $SO_4^{=}$	0.2 to 6.7 μg/mL	
OSHA[f] Method ID-182	Air	Nitrogen Dioxide determined as nitrite (NO_2^-)		
OSHA Method ID-190	Air	Nitric Oxide determined as nitrite (NO_2^-)		

Table 4-6 (continued)
Summary of Ion Chromatographic Compliance Testing Methods

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[a] U.S. Environmental Protection Agency.

[b] For drinking water, surface water, mixed domestic and industrial waste water, ground water, reagent water, solids, and leachates.

[c] For disinfection byproducts in drinking and reagent water.

[d] Minimum detection limit (MDL) obtained with reagent water is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value exceeds zero.

[e] National Institute of Occupational Safety and Health.

[f] Occupational Safety and Health Administration.

Table 4-7
Examples of Commercial Quality Control Standards for Anion and Cation Analysis

Constituent Elements and Parameters	Concentration Unit	National Institute of Standards and Technology[a] Simulated Rain Water Standards		Environmental Research Associates Waste Water Quality Control Standards	
		SRM 2694-I	SRM 2694-II	ERT Certified	Advisory Range
Flouride	μg/mL	0.050 ± 0.002	0.098 ± 0.007	13.5[c]	11.6–15.4[c]
Chloride	μg/mL	(0.24)	1.0	243[c]	224–262[c]
Nitrate	μg/mL	NA	7.06 ± 0.15[b]	NA	NA
Nitrate plus Nitrite	μg/mL	NA	NA	10.4[d]	9.4–11.4[d]
Phosphate	μg/mL	NA	NA	4.0[d]	3.6–4.4[d]
Sulfate	μg/mL	2.73 ± 0.05	10.9 ± 0.2	249[c]	224–274[c]
Sodium	μg/mL	0.205 ± 0.009	0.419 ± 0.015	230[c]	207–253[c]
Magnesium	μg/mL	0.024 ± 0.002	0.051 ± 0.00	NA	NA
Potassium	μg/mL	0.052 ± 0.07	0.106 ± 0.008	230[c]	207–253[d]
Calcium	μg/mL	0.014 ± 0.003	0.049 ± 0.011	NA	NA
Ammonium	μg/mL	NA	1.0	NA	NA
Ammonia	μg/mL	NA	NA	7.9[d]	6.9–8.9[c]
Acidity	μg/mL	0.030 ± 0.002	0.284 ± 0.003	NA	NA
Conductivity at 29.0 °C		26 ± 2	130 ± 2	NA	NA
pH at 25 °C		4.27 ± 0.03	3.59 ± 0.02	9.1[c]	8.9–9.3[c]

Table 4-7 (continued)
Examples of Commercial Quality Control Standards for Anion and Cation Analysis

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[a] The certified values are based on proven reliable methods of analysis. The estimated uncertainties are 2 standard deviations of the certified values except for uncertainties associated with sulfate, acidity, pH, and specific conductance which are based on scientific judgment and are roughly equivalent to 2 standard deviations of the certified value.

[b] The nitrate value is not certified because of instability. It is believed that bacterial or fungal activity contributes to that instability.

[c] Minerals WasteWatR^{TM}.

[d] Nutrients WasteWatR^{TM}.

Table 4-8
Carbon Analysis Method Characteristics

Species Measured	Measurement Method	Measurement Principle
Total Carbon	Thermal Combustion (McCarthy and Moore, 1952; Belsky, 1971; Mueller et al., 1971, 1989; Patterson, 1973; Appel et al., 1976, 1979; Kukreja and Bove, 1976; Merz, 1978; Macias et al., 1979; Malissa, 1979; Pierson and Russell, 1979; Cadle et al., 1980a-b, 1983; Pimenta and Wood, 1980; Rosen et al., 1980; Novakov, 1981, 1982; Cadle and Groblicki, 1982; Muhlbaiser and Williams, 1982; Stevens et al., 1982, 1990; Tanner et al., 1982; Wolff et al., 1982; Groblicki et al., 1983; Countess, 1990; Hering et al., 1990)	Combustion or decomposition of a filter sample deposit to carbon dioxide followed by nondispersive infrared, gas chromatography (GC) with thermal conductivity detection, coulometry, or by GC with flame ionization detection (FID) following hydrogenation to methane.
Total Carbon, Organic Carbon, Elemental Carbon	Solvent Extraction Method (Brachaczek and Pierson, 1974; Gordon, 1974; Grosjean, 1975; Daisey et al., 1981; Japar et al, 1984)	A filter is extracted in an organic solvent by Soxhlet extraction to remove organic material. The extractable mass, organic carbon, is determined gravimetrically by weighing the filter before and after sample extraction. The unextractable carbon can be analyzed by the thermal combustion method to determine elemental carbon.
Total Carbon, Organic Carbon, Elemental Carbon, Carbonate Carbon	Thermal Manganese Oxidation (TMO) Method (Mueller et al., 1982; Fung, 1990)	Manganese oxide (MnO_2) is used as an oxidizing agent, present and in contact with the sample punches, throughout the analysis. Temperature changes distinguish between organic carbon (OC) and elemental carbon (EC). Carbon evolved at 525°C is classified as OC, and carbon evolved at 850°C is classified as EC. Carbonate carbon can be determined by acidification of the filter punch at room temperature.

Table 4-8 (continued)
Carbon Analysis Method Characteristics

Species Measured	Measurement Method	Measurement Principle
Total Carbon, Organic Carbon, Elemental Carbon, Carbonate Carbon (cont.)	Thermal/Optical Reflectance (TOR) or Thermal/Optical Transmittance (TOT) Method (Chow et al., 1993b; Adams et al., 1989c; Huntzicker et al., 1982, 1986; Johnson and Huntzicker, 1979; Johnson et al., 1980, 1981; Johnson, 1981; Shah, 1981, 1988; Shah et al., 1984, 1986; Shah and Rau, 1991; Sunset Laboratory, 1990; Watson et al., 1994b)	A filter punch is submitted to volatilization at temperatures of 120, 250, 450, and 550°C in a 100% helium atmosphere, then to combustion at temperatures of 550, 700, and 800°C in a 2% oxygen and 98% helium atmosphere. The carbon evolved at each temperature is converted to methane by a methanator and quantified by a FID. The reflectance or transmittance from the filter punch is monitored throughout the analysis to correct for the pyrolysis of organic material. OC is defined as that which evolves prior to re-attainment of the original reflectance or transmittance, and EC is defined as that which evolves after the original reflectance or transmittance has been attained. Carbonate carbon can be determined by acidification of the filter punch at room temperature.

Table 4-8 (continued)
Carbon Analysis Method Characteristics

Species Measured	Measurement Method	Measurement Principle
Absorbance (light absorbing carbon)	Optical Absorption, Transmission Densitometry (Bergstrom, 1973; Lin et al., 1973; Smith et al., 1975; Rosen and Novakov, 1977, 1978; Rosen et al., 1979, 1982; Weiss et al., 1979; Delumyea et al., 1980; Edwards, 1980; Jennings and Pinnick, 1980; Campillo, 1981; Cowen et al., 1981; Japar et al., 1981, 1986, 1990; Bergstrom et al., 1982; Clarke, 1982, 1983; Clark and Waggoner, 1982; Gerber, 1982; Gerber and Hindman, 1982; Hansen et al., 1982; Heintzenberg, 1982; Reagan, 1982; Twomey and Huffman, 1982; Clarke et al., 1984, 1987; Gundel et al., 1984; Clarke and Charlson, 1985; Foot and Kilsby, 1989; Chylek and Hallett, 1992; Horvath, 1993a-b)	The light transmittal through a filter before and after sampling is measured. This method assumes that elemental carbon is the only light absorbing species on the filter. Empirically derived absorption coefficients are required for different filter media to estimate elemental carbon concentrations.

Table 4-8 (continued)
Carbon Analysis Method Characteristics

Species Measured	Measurement Method	Measurement Principle
	Integrating Plate or Integrating Sphere Method (Clarke, 1982; Campbell et al., 1989)	The Integrating Plate method measures the light that is transmitted in a forward cone before and after sample collection. The decrease in intensity for the exposed filter is assumed to be caused by optical absorption. The underlying assumptions are: (1) the amount of light reaching the detector is not affected by particle scattering; (2) no internal reflections within the filter will interact with absorbing particles; (3) no interference between particles (mass corrections are applied to correct for interference). For the Integrating Sphere method, the exposed filter is first placed at the front of the sphere to measure the amount of transmitted light, then moved to the back of the sphere to measure the reflected light. Large angle scattering by particles and internal scattering in the filter do not affect the measurement in this method.

Table 4-9
Sampling and Analysis Methods for Common Ambient VOC Classes

Common VOC Class	Most Applicable Sampling Method	Other Sampling Methods	Reference Method	Comments
HYDROCARBONS:				
Methane	Canisters		Tang et al., 1993	See also Des Tombe et al., 1991, for C_1–C_4 hydrocarbon sampling and analysis method review
C_2–C_3	Canisters	Carbon molecular sieves	U.S. EPA, 1991	
C_4–C_{10}	Canisters	Carbotrap; Bags	U.S. EPA, 1991	
C_{11}–C_{20}	Tenax		Zielinska and Fung, 1992	
Semivolatile PAH	Filter/PUF	Filter/XAD; Filter/Tenax	EPA Method TO13 (U.S. EPA, 1988)	
CARBONYL COMPOUNDS	DNPH-coated C_{18} Sep-Pak	DNPH-coated Sep-Pak; Impingers; Cryogenic traps	Druzik et al., 1990	Cryogenic method: see Pierotti, 1990
ORGANIC ACIDS	Base-coated filters	Base-coated C_{18} Sep-Pak; Denuders; Mist chamber	Andreae et al., 1987	See Tanner et al., 1993, for critical review
HALOGENATED HYDROCARBONS	Canisters	Porous polymers; Multibed sorbents	EPA Method TO14 (U.S. EPA, 1988)	
ALCOHOLS C_1–C_4	Charcoal	Canisters; Impingers with water; Cryogenic traps; Condensation sampling	NIOSH Method[b] Manual (NIOSH, 1984)	The applicability of charcoal collection/CS_2 elution method for ambient oxygenated compounds is currently being evaluated by AeroVironment, Inc. (1992)
PHENOLS/CRESOLS	Impingers with 0.1 N NaOH Filter/PUF	Porous polymers	EPA Method TO8 (U.S. EPA, 1988) Hawthorne et al., 1988; 1989	

Table 4-9 (continued)
Sampling and Analysis Methods for Common Ambient VOC Classes

Common VOC Class	Most Applicable Sampling Method	Other Sampling Methods	Reference Method	Comments
ETHERS	Charcoal	Canisters; Porous polymers; Carbotrap; Cryogenic trapping	NIOSH Method Manual (NIOSH, 1984)	See comment for alcohols
ESTERS	Charcoal	Carbotrap; Porous polymers; Cryogenic trapping; Canisters	NIOSH Method Manual (NIOSH, 1984)	See comment for alcohols
ORGANIC NITROGEN COMPOUNDS:				
Nitrosamines	Porous polymers	Ascorbic acid solution	EPA Method TO7 (U.S. EPA, 1988)	See Brunneman et al., 1980, for ascorbic acid method
Amines C_1–C_4	Acidified C_{18} Sep-Pak	Porous polymers; Impingers with acidic solution	Kuwata et al., 1983a	
Aromatic Amines	Silica gel	Porous polymers; Impingers with acidic solution	Intersociety Committee, 1989b	
Alkyl Nitrates	Charcoal	Porous polymers	Atlas and Schauffler, 1991	
Nitro-aromatics	Porous Polymers[a]			
SEMIVOLATILE PESTICIDES	Filter/PUF	Filter/XAD; Filter/Tenax	EPA Methods TO4, TO10 (U.S. EPA, 1988)	
ORGANIC SULFUR GASES	Porous polymers	Canisters; Bags		

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[a] Including PUF.

[b] Silica gel is recommended for methanol (NIOSH Method 2000).

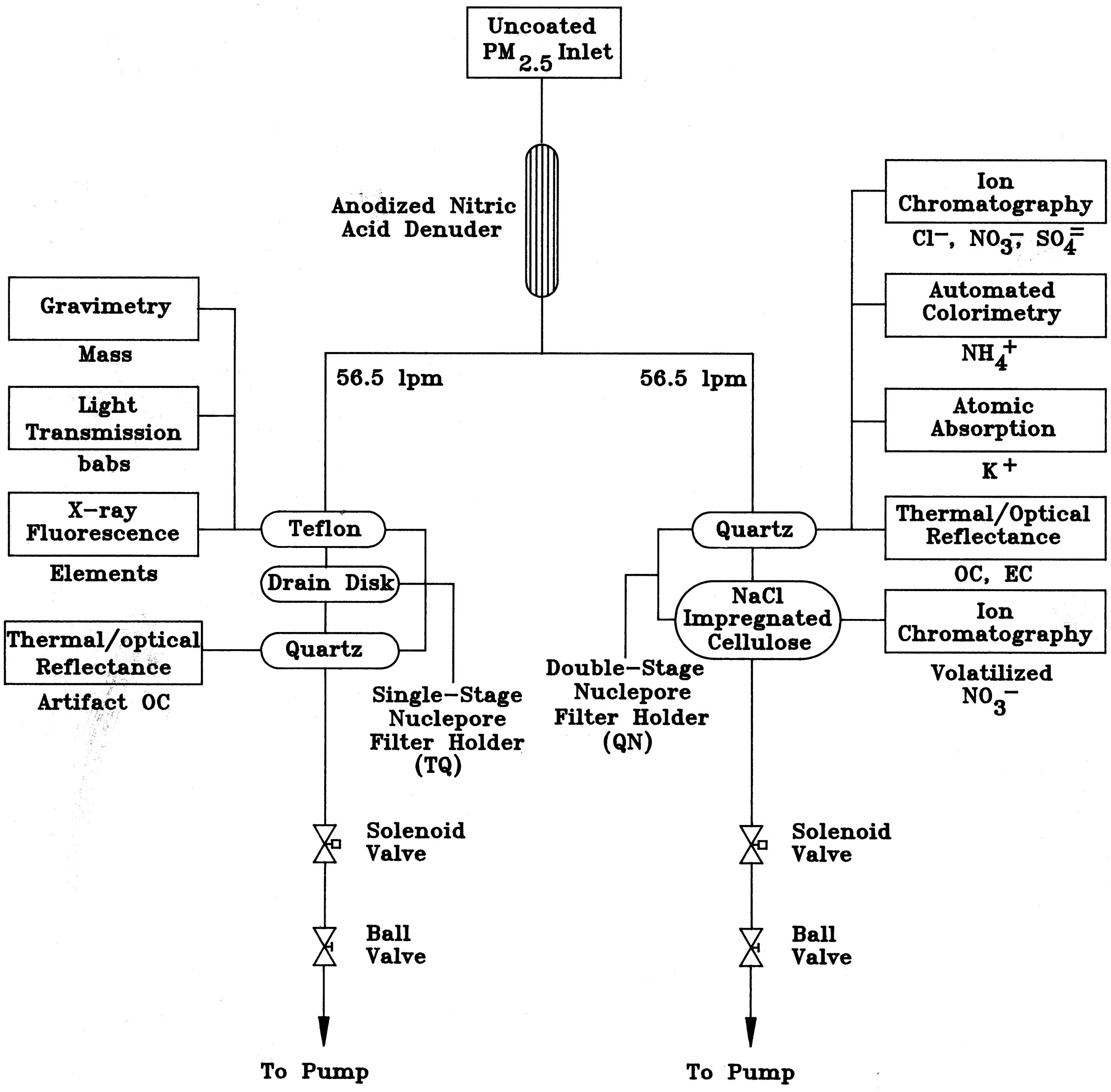


Figure 4-1. Flow diagram of the sequential filter samplers for $PM_{2.5}$ mass, light absorption, elements, ions, and carbon measurements at the Welby site during winter and summer 96 and the Brighton and Welby sites during winter 97.

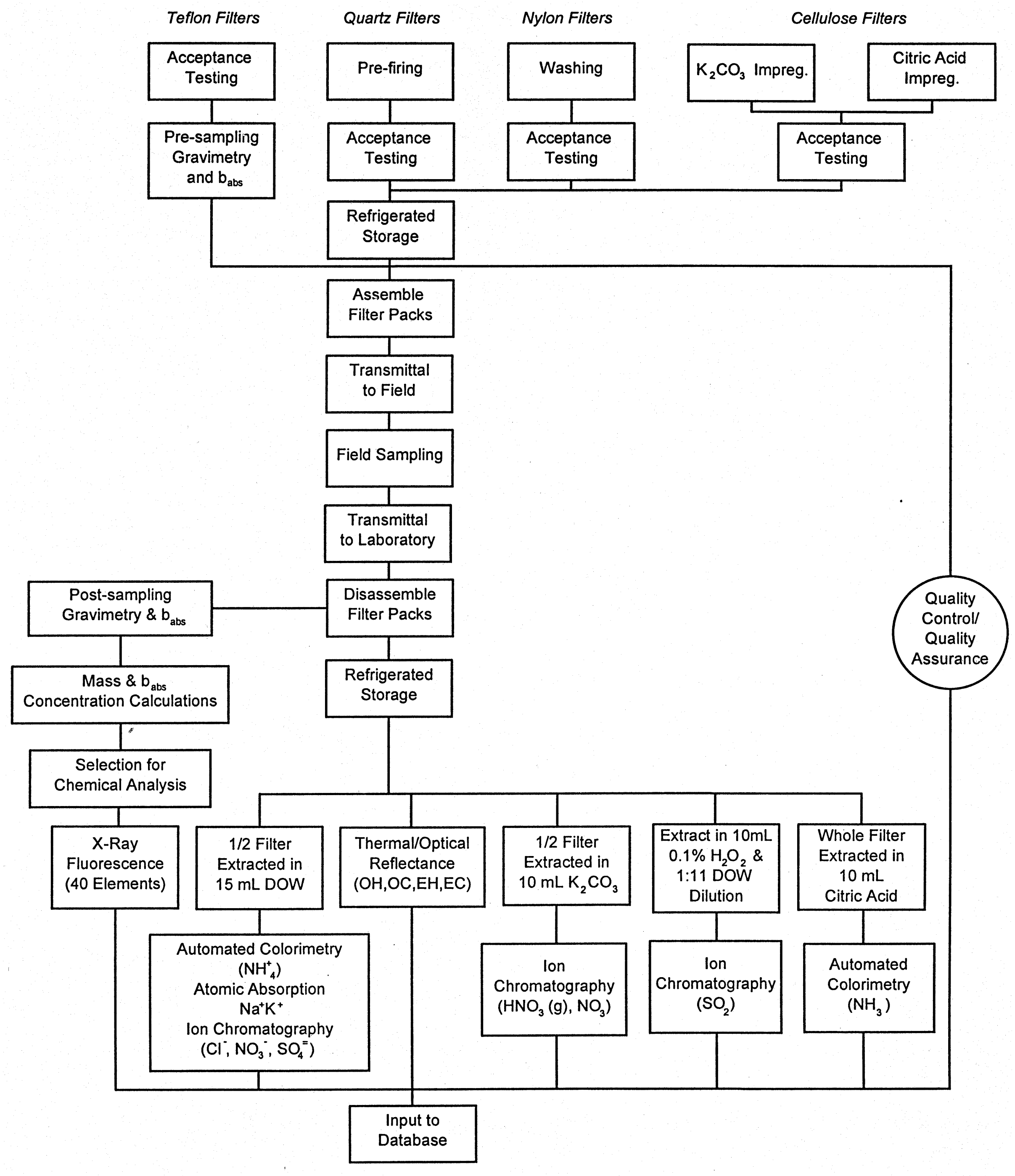


Figure 4-2. Flow diagram of filter processing and chemical analysis activities for the aerosol and gaseous sampling system.

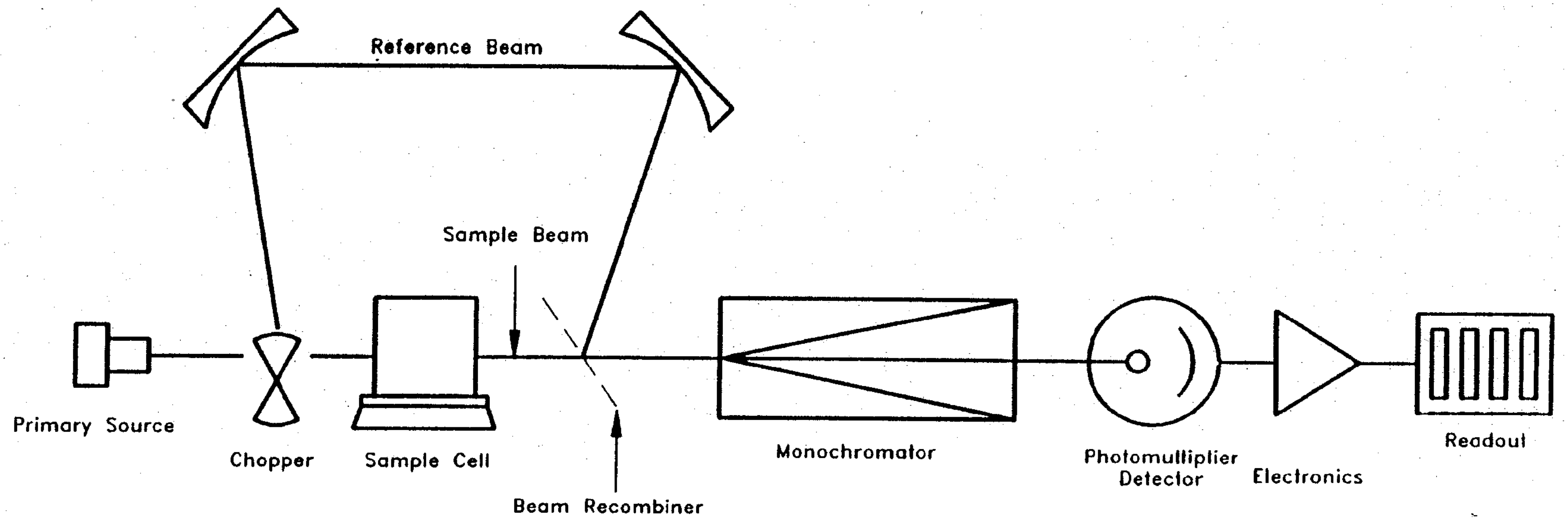


Figure 4-3. Schematic of a typical double-beam AAS system.

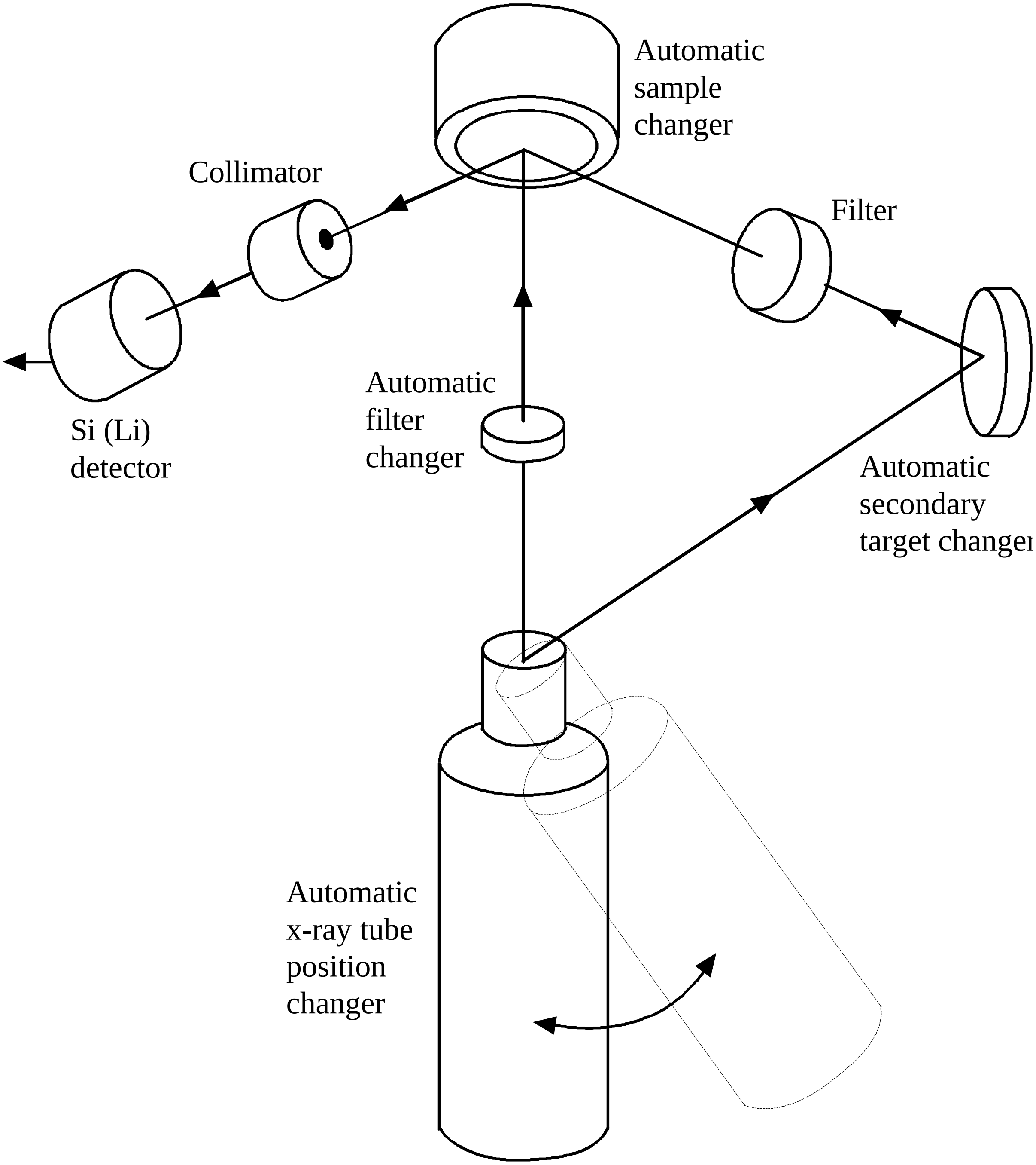


Figure 4-4. Schematic of a typical x-ray fluorescence (XRF) system.

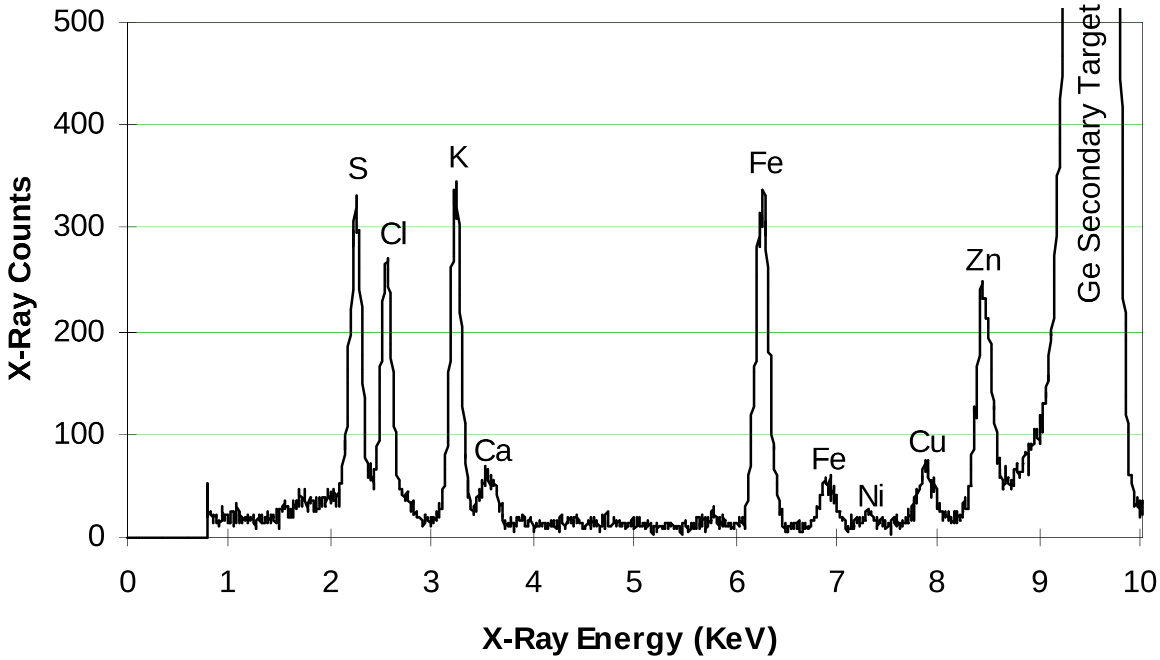


Figure 4-5. Example of an x-ray fluorescence (XRF) spectrum obtained under Condition 3.

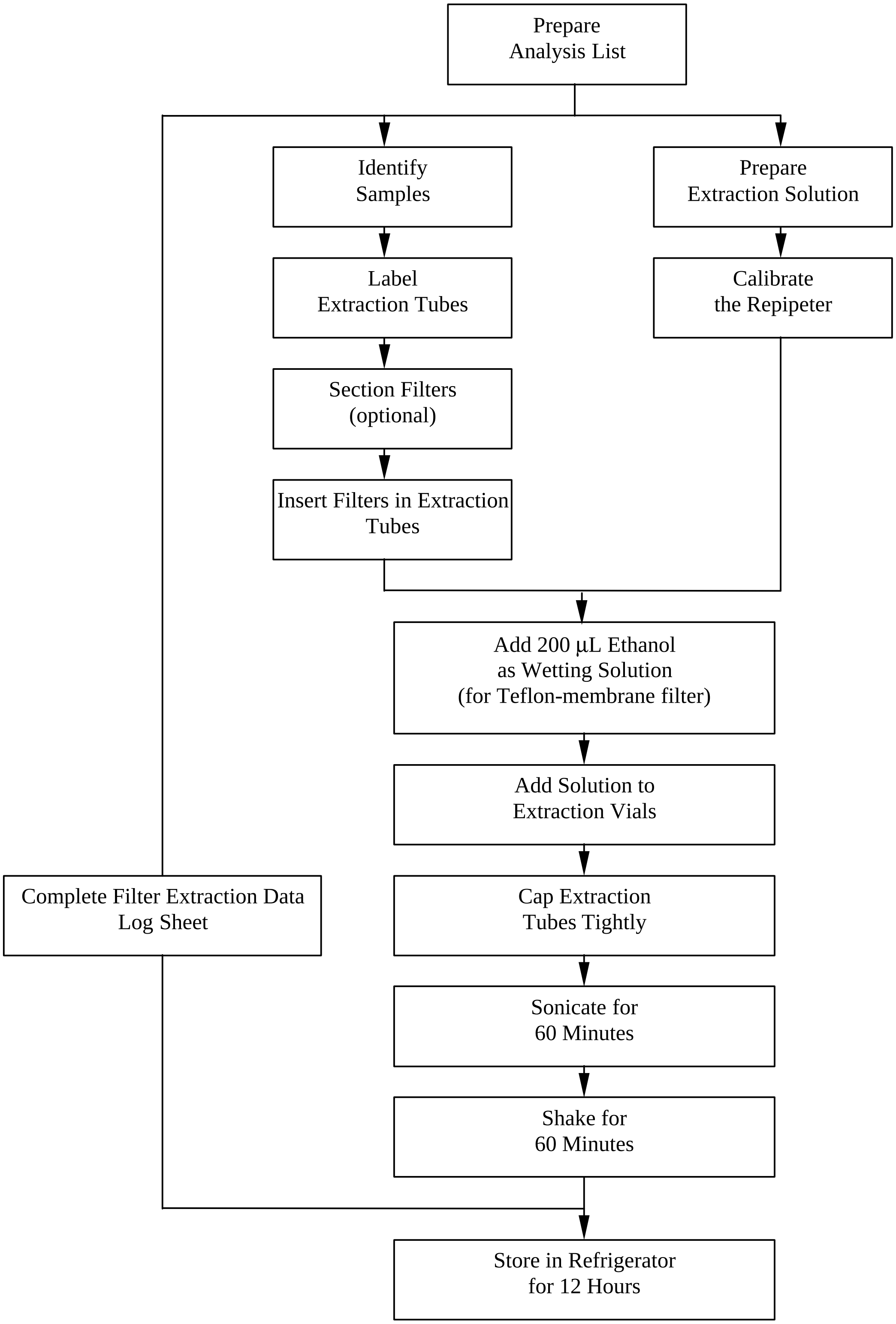


Figure 4-6. Flow diagram of filter extraction procedure.

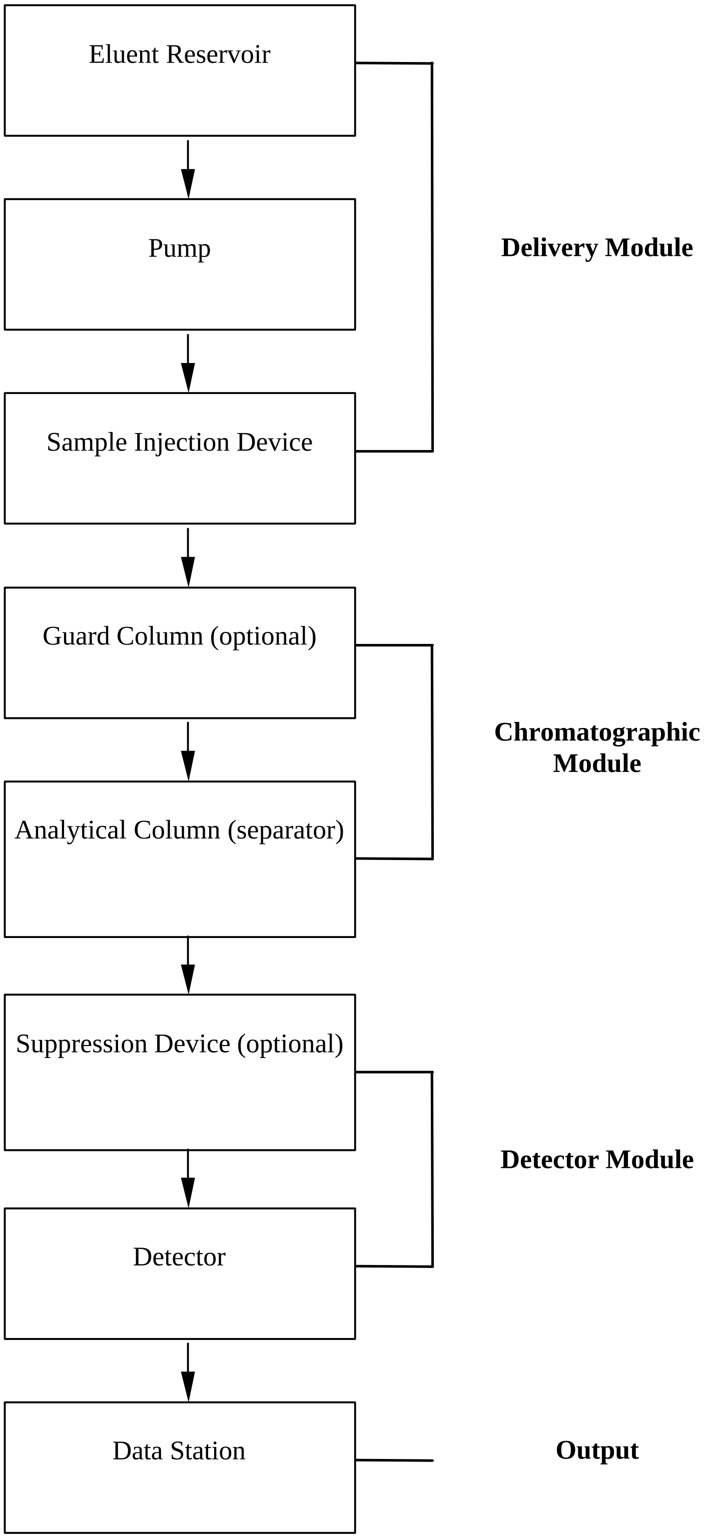


Figure 4-7. Basic components of the ion chromatography system.

```
=============================================================================
| Sample Name: STD-F                          Date: Mon Oct 16 12:14:09 1989|
| Data File  : A:\1018T1.D04                                                |
| Method     : C:\DX\METHOD\ANION1.MET                                      |
| ACI Address: 1        System : 1       Inject#:   4      Detector: CDM-2  |
=============================================================================
***********************  EXTERNAL STANDARD REPORT  **************************

Stop time =  5.02 Minutes           Number of Data Points =  3010
Area reject = 100           One Data Point per 0.1 seconds
Amount Injected = 1       Dilution factor = 1.

PEAK  RET    PEAK                CONC. in                              REF   % DELT
NUM   TIME   NAME                ug/ml         AREA       HEIGHT BL  PEAK   RET TI
-----------------------------------------------------------------------------
 1    1.55 CHLORIDE            4.775e-001  3.235e+007 6602913  1    1    0.00%
 2    1.85 NITRITE             4.885e-001  1.944e+007 3811841  1    2    0.00%
 3    3.17 NITRATE             4.914e-001  1.771e+007 2137115  1    3    0.00%
 4    4.20 SULFATE             4.908e-001  2.338e+007 2278689  1    4    0.00%
```

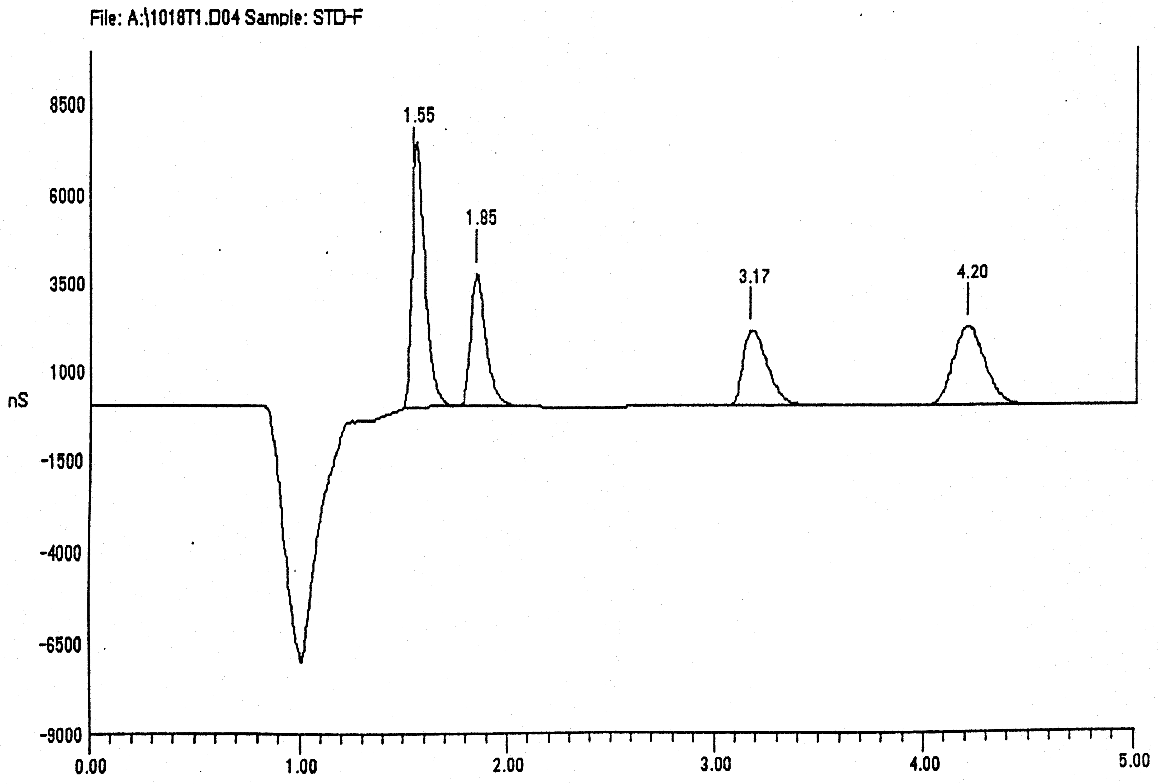


Figure 4-8. Example of ion chromatogram for calibration standard at 0.5 μg/mL.

Method: ANION2.MET
Component: Chloride
Fit Type: Linear
r^2: 0.999590
Conc = Resp * 1.73E–008 + 0.002335
Resp = Conc * 5.779E+007 + –1.349E+005
Standardization: External
Calibration: Area

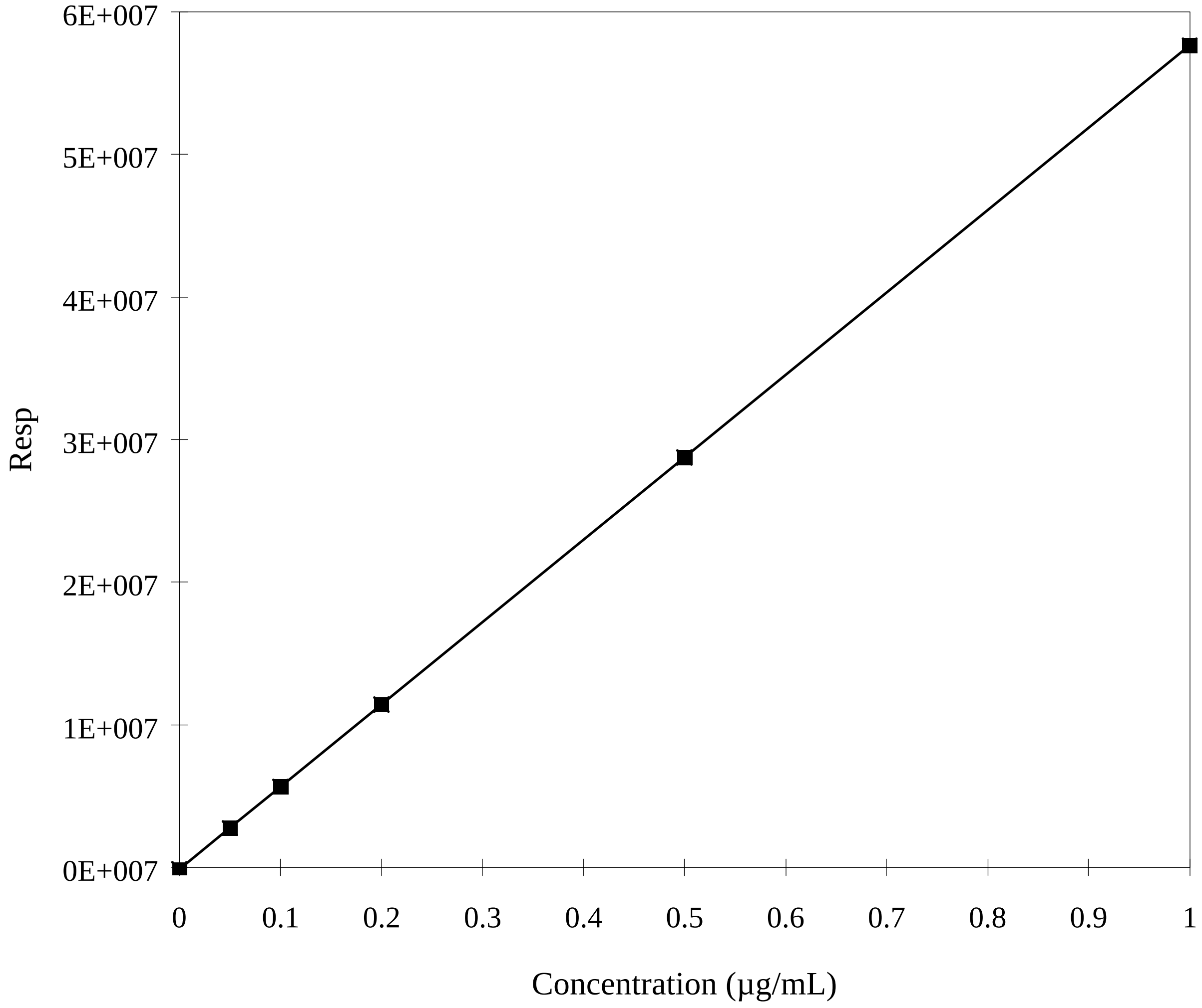


Figure 4-9. Example of ion chromatography calibration curve.

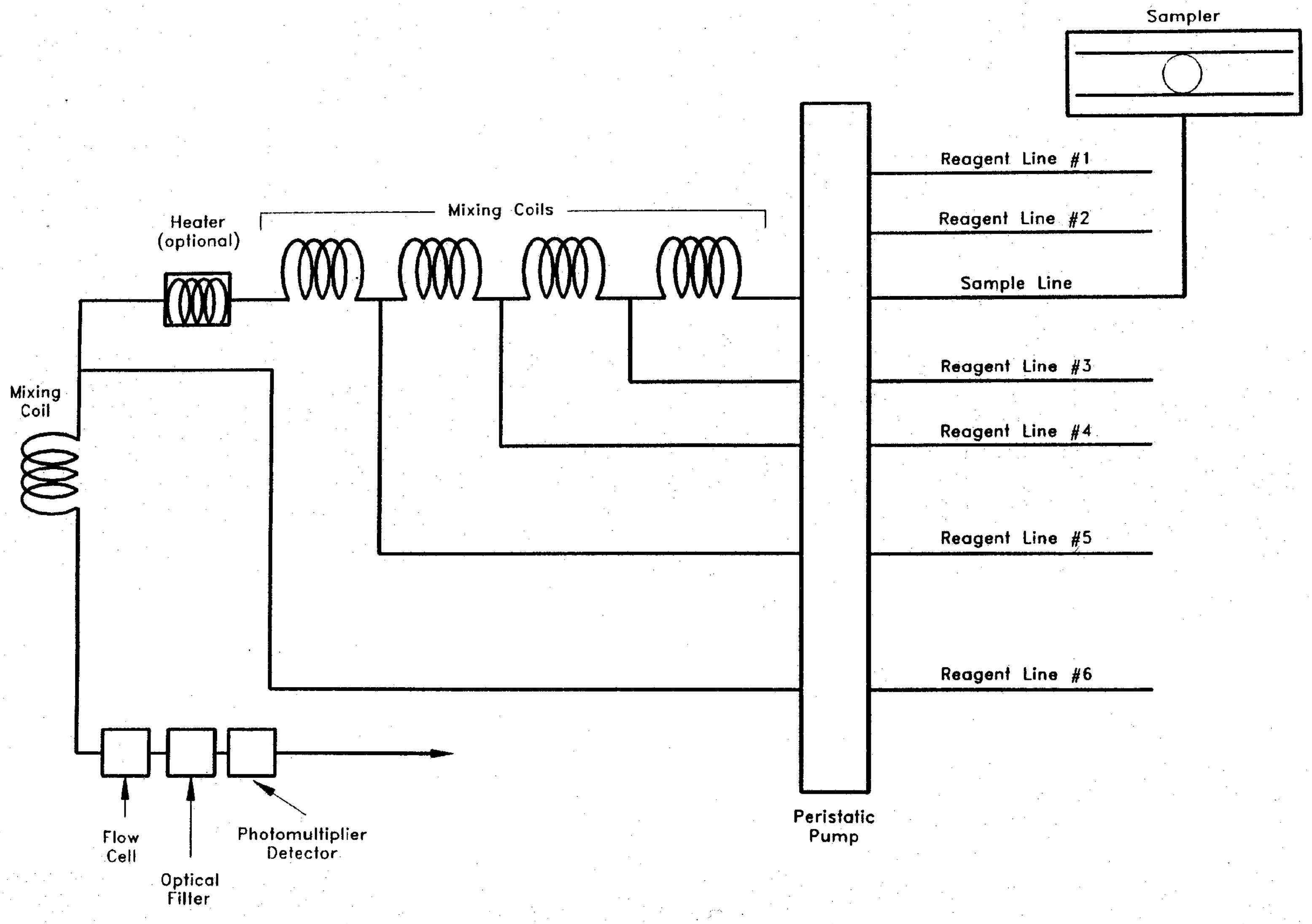


Figure 4-10. Schematic of a typical automated colorimetry (AC) system (Technicon Instruments Corporation, 1986).

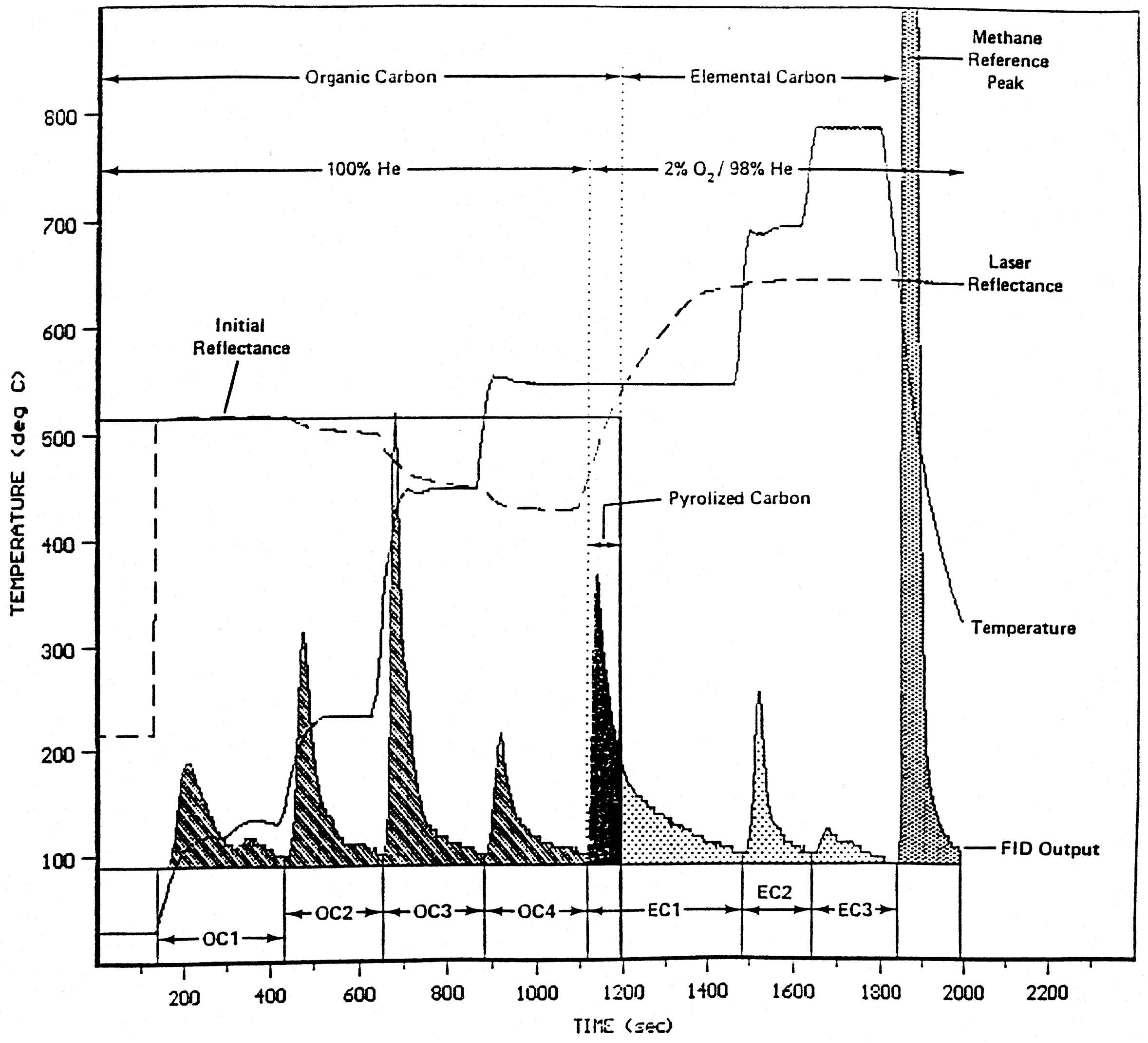


Figure 4-11. Example of a thermal optical reflectance (TOR) thermogram (Chow et al., 1993).

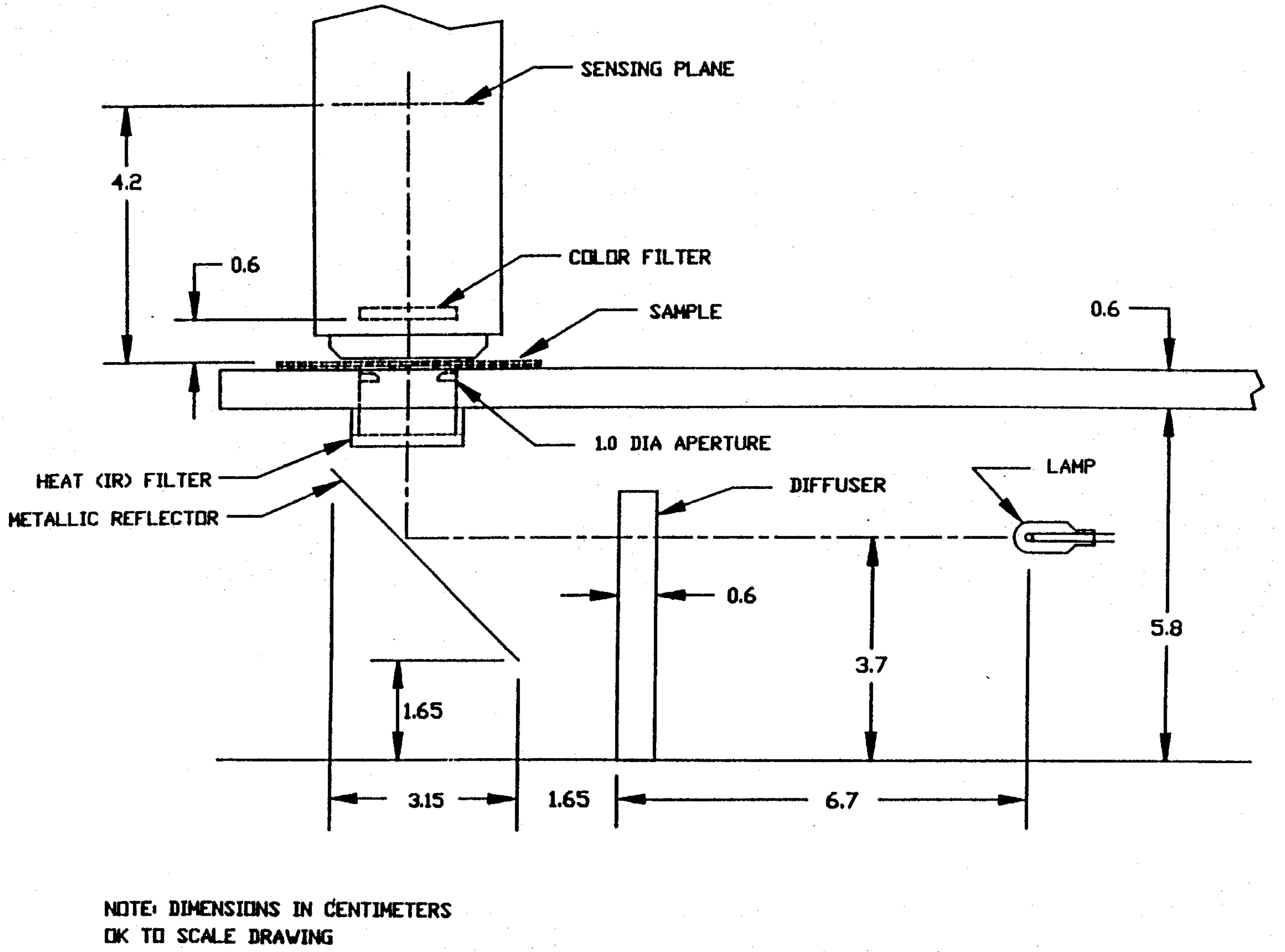


Figure 4-12. Example of a light transmission measurement system (Watson et al., 1988b).

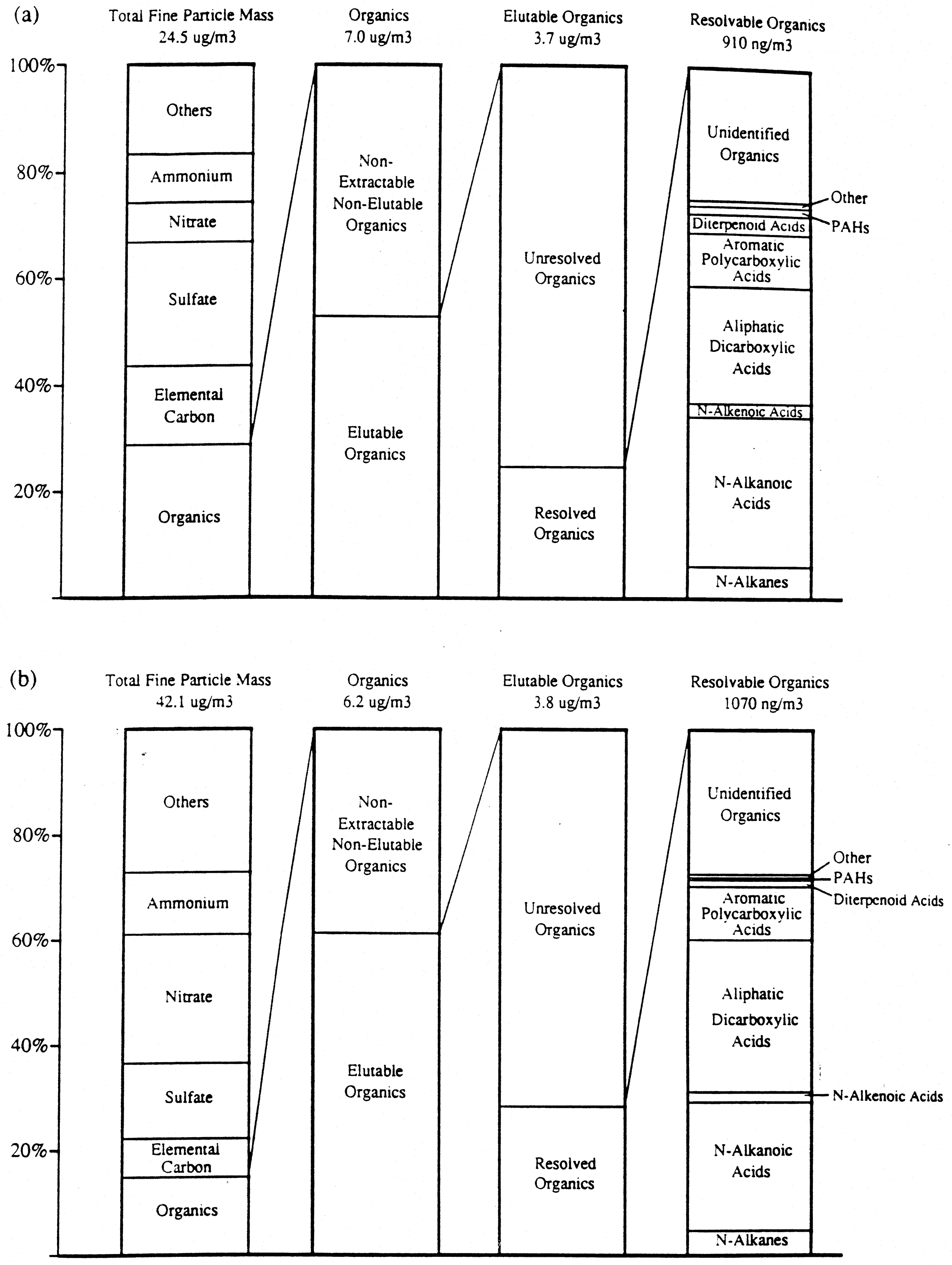


Figure 4-13. Mass balance on the chemical composition of annual mean fine particle concentrations (1982) for West Los Angeles and Rubidoux (Riverside), CA (from Rogge et al., 1993).

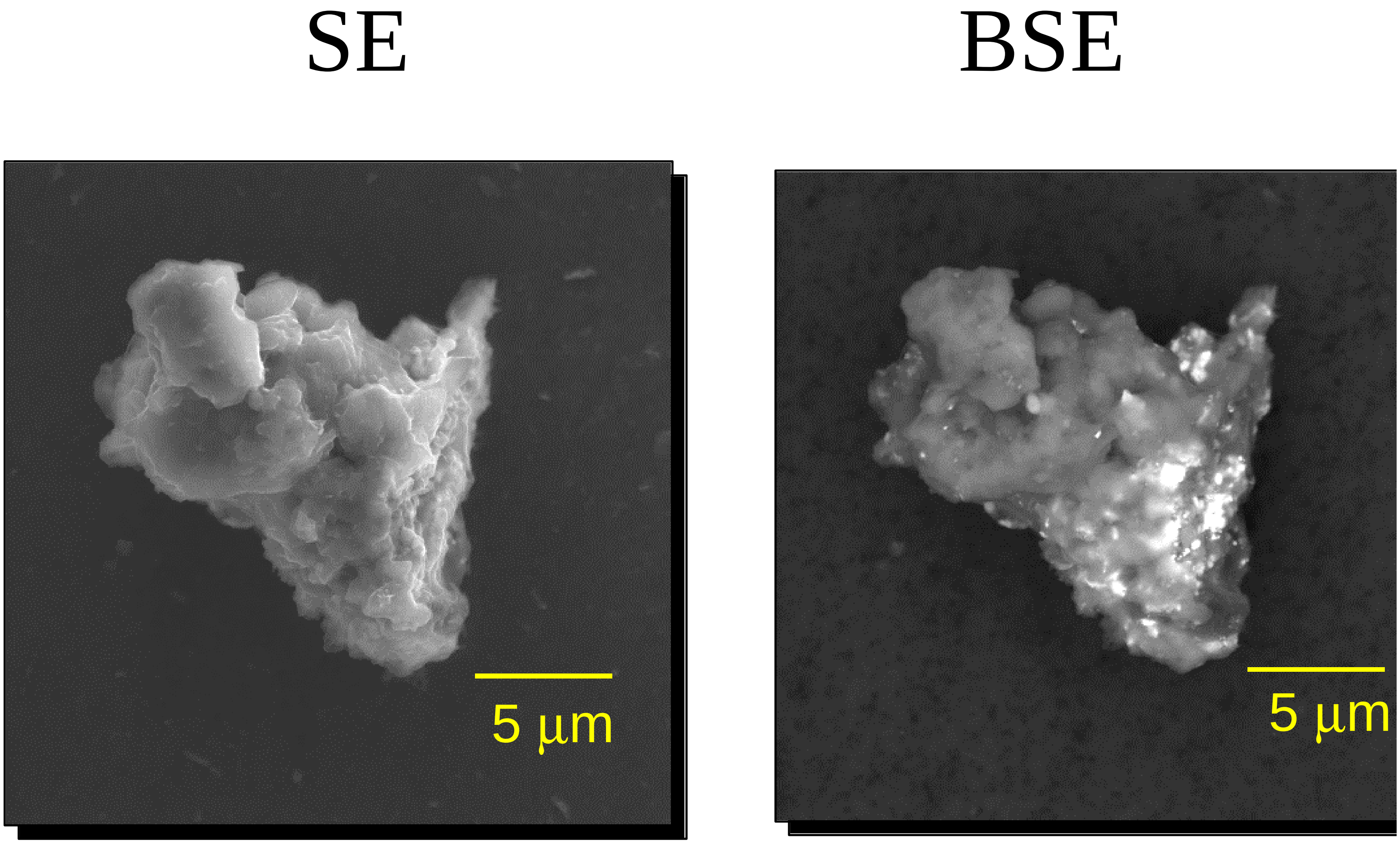


Figure 4-14. Example of secondary electron image (SE) and backscattered electron image (BSE) of a particle from scanning electron microscopy analysis.

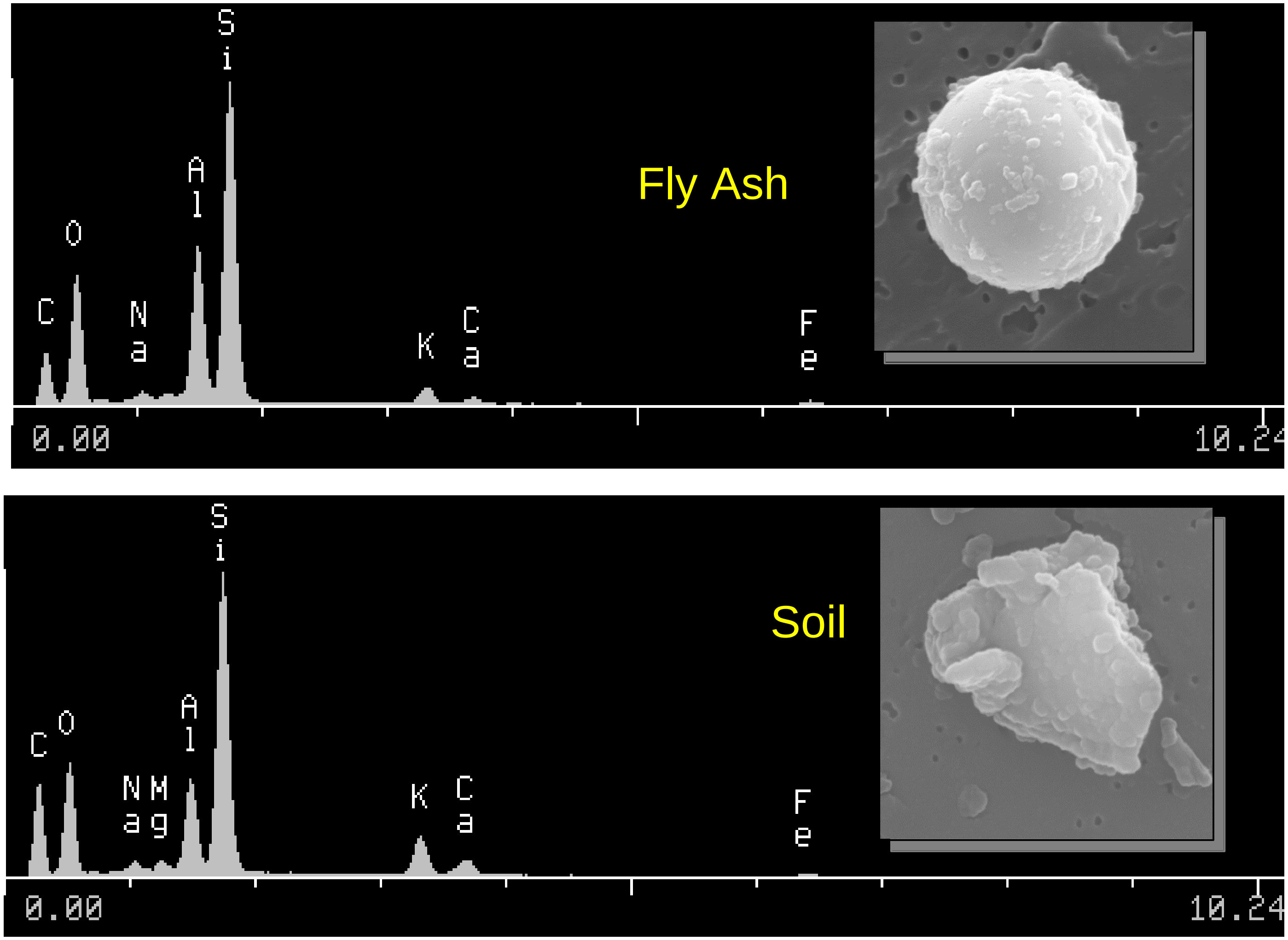


Figure 4-15. Example of elemental information on fly ash and soil particles obtained from computer-controlled scanning electron microscopy.

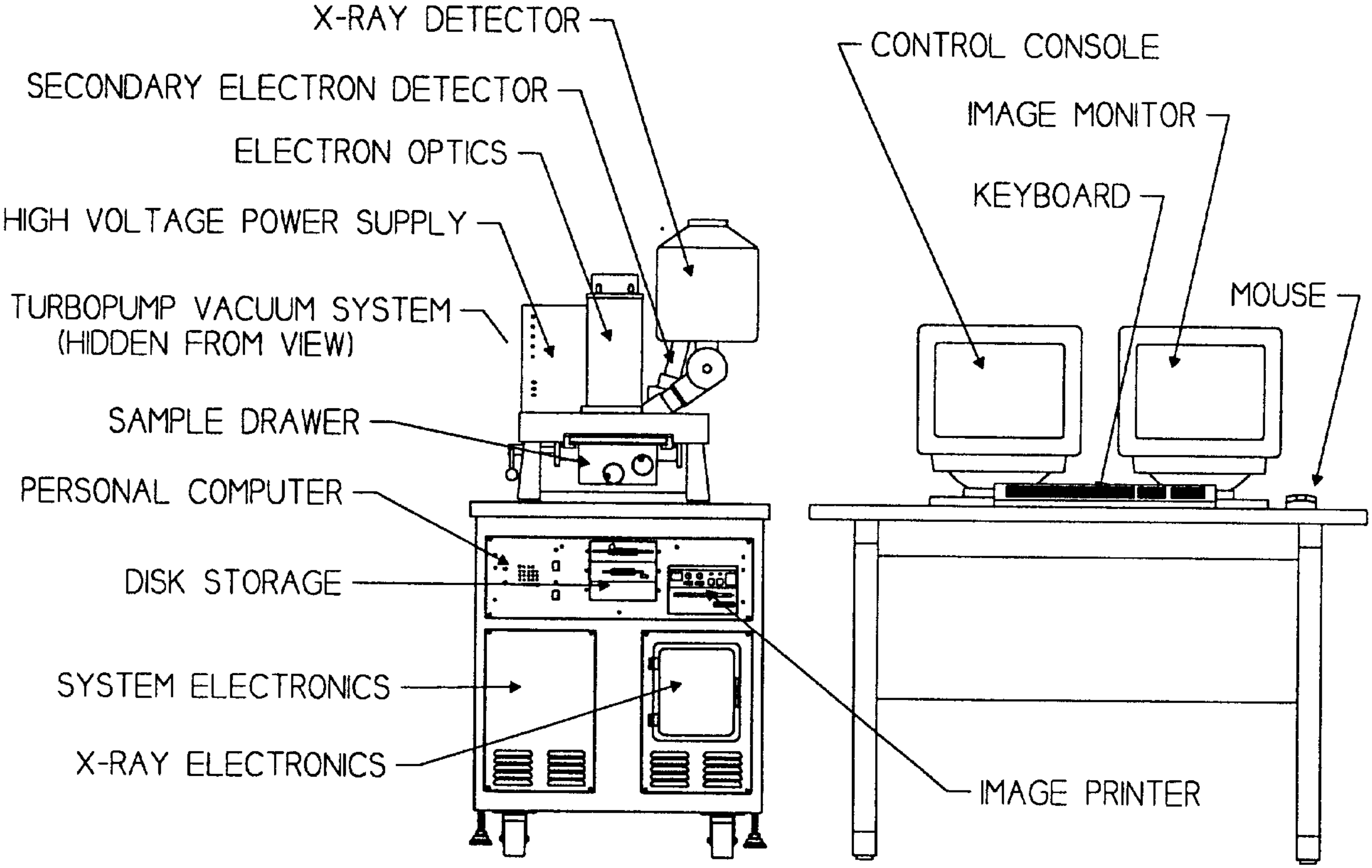


Figure 4-16. Schematic of the personal scanning electron microscopy system.

5.0 MEASUREMENT ARTIFACTS AND INTERFERENCES

This section discusses some of the general difficulties in aerosol measurement with respect to: (1) particle and gas removal in inlets; (2) nitrate particle volatilization; (3) adsorption of sulfur dioxide and oxides of nitrogen; (4) organic carbon adsorption and volatilization; (5) liquid water content; (6) electrostatic charging; and (7) passive deposition and recirculation. It demonstrates that some of these biases can be avoided or counteracted with alternative sampling and analysis techniques. It also identifies areas where more research is needed to better explain or eliminate these biases.

5.1 Particle and Gas Removal in Inlets

The materials from which inlets are manufactured can affect collected aerosol, especially when gas and particle phases are being quantified for volatile species such as ammonium nitrate. Most samplers are manufactured from aluminum, plastic, or galvanized steel owing to their availability and economy. These materials can absorb some gases (Henry et al., 1988; John et al., 1988; Soderholm, 1995), especially nitric acid, that can change the equilibrium of volatile particles on a filter with the surrounding air (Biswas et al., 1987, 1990).

John et al. (1986) and Fitz and Hering (1996) show that surfaces coated with perfluoroalkoxy (PFA) Teflon can pass nitric acid with 80% to 100% efficiency. They also show that the aluminum surfaces common to many samplers and inlets have an almost infinite capacity for absorbing nitric acid vapor while transmitting particles with high efficiency (John et al., 1988). Plastic surfaces can acquire an electrical charge which might attract suspended particles, though the dimensions of most ambient sampling systems are sufficiently large that this attraction is negligible (Rogers et al., 1989).

Denuders (Biswas et al., 1990) are often used as part of or immediately behind size-selective inlets to remove gases that might interfere with the aerosol measurement, or to quantify the concentrations of gases that are precursors to secondary aerosols. Denuders take advantage of the fact that gas molecules diffuse through air much more rapidly than small particles.

Denuder surfaces are made of or coated with substances that absorb the gases of interest. When properly coated, the denuder surfaces can be washed and the solvent analyzed for the absorbed gases. Denuder geometries can be rectangular, cylindrical, or annular; the annular designs provide the highest gas collection efficiency so their lengths can be minimized. When the walls of the denuder are coated with substances that absorb the gases, the denuders can be washed and the extract can be submitted to chemical analysis. This method is often used to measure nitric acid, sulfur dioxide, and ammonia gases along with suspended particles. These gases are common precursors to secondary sulfate and nitrate compounds.

5.2 Ammonium Nitrate Volatilization

Nitrate losses during and after sampling have been reported in numerous measurement programs (Dunwoody, 1986; Witz et al.,1990). Russell et al. (1983) estimate at temperatures greater than 30 °C, most nitrate will be in the gas phase as nitric acid (HNO_3), while at temperatures lower than 15 °C most nitrate will be in the particle phase as ammonium nitrate (NH_4NO_3), and that there will be varying amounts of nitric acid and ammonium nitrate for the intermediate temperatures. Relative humidity and concentrations of ammonia and nitric acid gases also affect this equilibrium, but temperature is by far the most important variable. When air temperatures changes during sampling, some of the ammonium nitrate already collected on the filter can volatilize.

This volatilization artifact is illustrated in Figures 5-1a and 5-1b for particulate nitrate. In these figures, the Total Particulate Nitrate was determined by sampling through a nitric acid denuder onto a quartz-fiber filter backed by a nylon-membrane filter; both filters were analyzed for nitrate. The Filter Nitrate was measured from a quartz-fiber filter drawn through the same $PM_{2.5}$ inlet with a denuder but without a backup filter to adsorb the volatilized particulate nitrate. Figure 5-1a shows samples were taken throughout the day during summer in California. Temperatures ranged from ~5 °C to >30 °C during these experiments. There are many cases where the Filter Nitrate is less than 50% of the Total Particulate Nitrate.

There are also many cases, however, where there is agreement between the two measurements. Good agreement and poor agreement are found for nighttime, low-temperature samples as well as for afternoon, high-temperature samples. There is still a poor understanding of how well volatile compounds can be accurately measured by filter sampling.

Nitrate volatilization is minimized when ambient temperatures are low. As shown in Figure 5-1b, very little or no volatilized nitrate was found during the winter study period. The average ratio of Filter Nitrate (i.e., non-volatilized nitrate) to Total Particulate Nitrate (i.e., non-volatilized plus volatilized nitrate) in either the PM_{10} or $PM_{2.5}$ fractions was less than 5%, which is well within the measurement errors.

These comparisons demonstrate that nitrate volatilization was not significant (in the range of 5% to 10%) during the fall and winter studies. They also show that gaseous nitric acid levels were low throughout the fall and winter study regions. These volatilized nitrate values were well within the ±10% measurement uncertainties. It appears that reasonably accurate and precise particle nitrate concentrations were obtained in these study regions during fall and winter without complex denuder systems. Volatilized nitrate was not part of the measured $PM_{2.5}$ or PM_{10} mass, so this loss does not affect $PM_{2.5}$ or PM_{10} mass concentrations.

Volatile compounds can also leave the filter after sampling and prior to filter weighing or chemical analysis. Witz et al. (1990) observed greater than 50% losses of nitrate, ammonium, and chloride from glass- and quartz-fiber filters that were stored in unsealed containers at ambient air temperatures for two to four weeks prior to analysis. Refrigerating filters in sealed containers will minimize such losses.

5.3 Sulfur Dioxide and Oxides of Nitrogen Adsorption

PM measurements on glass fiber filters are biased by adsorption of sulfur dioxide, oxides of nitrogen, and nitric acid owing to filter alkalinity (Lee and Wagman, 1966; Coffer et al., 1974; Meserole et al., 1976, 1979; Pierson et al., 1976, 1980; Coutant, 1977; Spicer and Schumacher, 1977; Witz and McRae, 1977; Watson et al., 1981; Witz and Wendt, 1981a, 1981b, 1985; Appel et al., 1984; Lipfert and Wyzga, 1995). These result in positive biases that can be as high as 10 $\mu g/m^3$. Such biases are largely eliminated by the filter acceptance requirement of alkalinity less than 25 micro-equivalents per gram, typical of Teflon-membrane and quartz-fiber materials.

5.4 Organic Carbon Adsorption and Volatilization

Two issues are often confused when discussing filter contamination: gas adsorption (positive artifact) and particle volatilization (negative artifact). Adsorbed gases result from adherence of gases to the filter medium and result in positive biases to mass and chemical concentration. Gas adsorption depends on the filter material. Some particles change to gases, or volatilize, when temperatures, relative humidities, and precursor gas concentrations change during sampling or during handling and storage. Volatilization causes a negative bias to mass and chemical composition and is more dependent on environmental variables than on the filter composition. The velocity with which air is drawn through a filter can affect both the adsorption and volatilization properties of particles and gases (McDow and Huntzicker, 1990).

Organic vapors are adsorbed by quartz-fiber filters, but semi-volatile organic particles also evaporate during sampling and storage. Both negative and positive biases to $PM_{2.5}$ mass and organic carbon measurements result from these competing processes, and it is not yet known which ones dominate in a particular environment.

Eatough et al. (1989, 1990) and Tang et al. (1994) concluded that desorption of organic gases from particles on the front quartz-fiber filter was the dominant sampling artifact (negative bias), while Turpin et al. (1994) found the adsorption of organic gases by quartz-fiber filters (positive bias) was the prevalent interferent for mass and organic carbon concentrations on this material (McMurry and Zhang, 1989).

Turpin et al. (1994) found that adsorbed organic gases constituted up to 50% of the organic carbon measured on quartz filters in southern California. Their studies concluded that: (1) organic gas adsorption (positive bias) was much larger than organic particle volatilization (negative bias); (2) a quartz-fiber filter located behind a Teflon-membrane filter can quantify the adsorbed gases; (3) as sample durations increase, the proportion of the adsorption bias decreases because the adsorbed gases reach equilibrium with the collected particles and the filter can become saturated; and (4) the composition of the organic gases and particles in the atmosphere affect the magnitude of the artifact.

Chow et al. (1996a) compared the organic carbon measured on the backup quartz-fiber filter behind the front Teflon-membrane filter with the $PM_{2.5}$ particulate organic carbon measured on the front quartz-fiber filter which sampled alongside this filter pack. It was found that organic carbon concentrations on the backup filter were frequently 50% or more of the front quartz-fiber filter concentrations at all ten non-urban and regional sampling locations in California's San Joaquin Valley during ozone episodes. If the backup filter only quantifies organic gases that are adsorbed on the quartz-fiber filter, then the backup filter concentrations should be subtracted from the front filter concentrations, as suggested by Turpin et al. (1994). However, this subtraction resulted in negative organic carbon concentrations in 33% of 500 samples.

Chow and Egami (1997) reported that the extent of gaseous organic carbon adsorption was dependent upon the source mixture in the atmosphere. Figure 5-2 shows that the ratio of backup to front organic carbon becomes fairly constant at $PM_{2.5}$ concentrations greater than 30 $\mu g/m^3$, but the ratio varies and is frequently higher at lower $PM_{2.5}$ concentrations. This suggests the adsorbed gas reached equilibrium with the particles and adsorption sites on the front filter became saturated (Turpin et al., 1994). Similar relationships were also found during winter fog episodes in California's Central Valley (Chow and Egami, 1997) and during the winter in northwestern Colorado (Watson et al., 1996a).

5.5 Liquid Water Content

The importance of liquid water content in ambient aerosol has been recognized (Ho et al., 1974; Whitby and Sverdrup, 1980; Pilinis and Seinfeld, 1987). As ambient relative humidity exceeds 70%, particle growth becomes significant. Rogers and Watson (1996) summarize the physical chemistry necessary to estimate the deliquescent and efflorescent behavior of solution droplets. It was found that either theoretical calculation or experimental measurement of aerosol liquid content is complicated because growth rates vary with aerosol composition, ambient relative humidity, water activity, and surface tension (Hinds, 1982).

The current $PM_{2.5}$ NAAQS requires filter equilibrium within ±5% for relative humidity between 30% and 40% and within ±2 °C for temperature between 20 °C and 23 °C. Figure 5-3 gives an example of the effects of liquid water on particle mass concentrations within this equilibration range. These samples, which contain 43% ammonium nitrate and 57% sea salt, were collected in a coastal environment in Europe (Hanel and Lehmann, 1981). Figure 5-3 shows that as relative humidity approaches the 45% upper limit of the current equilibrium scale, mass concentrations can increase by 7% to 8%.

If samples were acquired in a very humid environment where soluble particles tend to aggregate in liquid water molecules, and then equilibrated in a laboratory environment where relative humidity is controlled at 25%, a difference of 10% or more in mass concentrations could occur depending on particle and filter composition (Demuynck, 1975; Charell and Hawley, 1981). When samples are acquired in an environment rich in sulfuric acid or ammonium sulfate, this bias could be as high as 15% to 20%. Therefore, to minimize the effect of liquid water on measured particles, it is recommended that relative humidity equilibration ranges be kept at the low end of 25% to 30% for filter weighing.

5.6 Electrostatic Charge

Static charge is the accumulation of electrical charges on a dielectric surface (such as the surface of a Teflon-membrane or polycarbonate-membrane filter). As discussed in Section 4.3, residual charge on a filter can produce an electrostatic interaction that induces non-gravimetric forces between the filter on the microbalance weighing pan and the metal casing of the electrobalance, thereby biasing the mass measurements (Engelbrecht et al., 1980; Feeney et al., 1984). As electrostatic charges build up during the weighing session, the readout of the microbalance becomes increasingly unstable (e.g., noisy, sudden drifts or shifts).

To minimize the static charge during gravimetric analysis, it is necessary to place a radioactive antistatic strip inside the microbalance chamber prior to weighing. Filters also need to be placed on an antistatic strip for at least 60 seconds to remove electrostatic charges (Hawley and Williams, 1974; Weil, 1991).

The most commonly used low-level radioactive antistatic strips contain 500 picocuries of polonium-210 (Po^{210}). Polonium strips are commercially available and have a half-life of 138 days. Polonium strips neutralize electrostatic charges on items within an inch of the strip surface. Antistatic solutions can also be used to coat the interior and exterior non-metallic surfaces of the weighing chamber. This coating increases surface conductivity and facilitates the draining of electrostatic charges from non-metallic surfaces to metallic surfaces.

Earth-grounded conductive mats should also be placed on the laminar flow hood where the balance is located or near the weighing table and equilibration chamber to reduce electrostatic charge buildup. This is especially important when weighing polycarbonate-membrane filters.

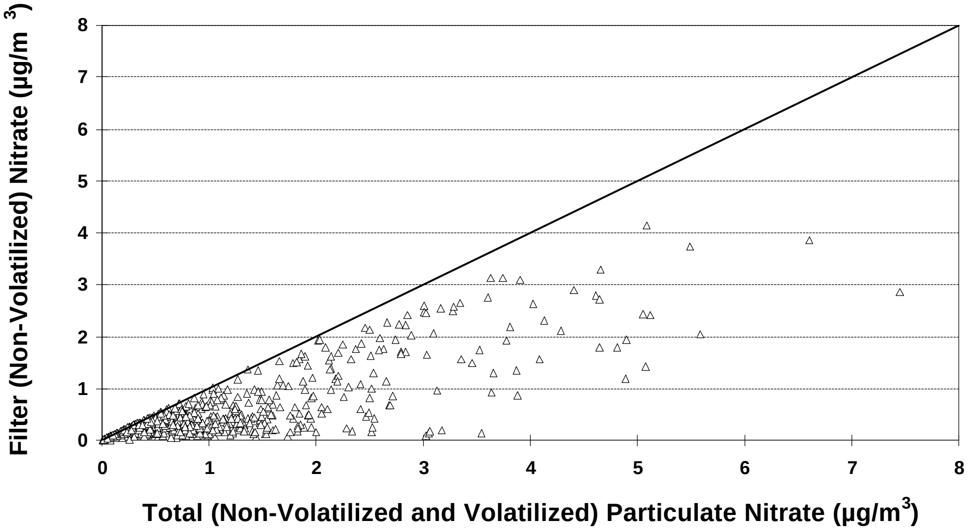
5.7 Passive Deposition and Recirculation

Passive deposition of windblown dust on the filter prior to and following sampling can positively bias PM measurements (Bruckman and Rubino, 1976; Chahal and Romano, 1976; Blanchard and Romano, 1978; Swinford, 1980). This bias can be minimized by more frequent sample changing (i.e., reduce the passive deposition period), pre-loading filters in the laboratory, and transporting unexposed and exposed filter cassettes in covered cassette holders.

Recirculation refers to the sampling of sampler pump exhaust (Countess, 1974; King and Toma, 1975). Recirculation does not have a large effect on $PM_{2.5}$, but it can effect carbon and trace metal measurements when pump and armature wear are entrained in pump exhaust. This contamination can be minimized by filtering pump exhausts or ducting them away from nearby sampling inlets. Even though $PM_{2.5}$ pumps may be adequately filtered, nearby high volume samplers for PM_{10} or TSP can still affect the $PM_{2.5}$ measurements and require filtration or ducting.

For $PM_{2.5}$ sampling, dynamic field blanks should be periodically placed in the sampling system without air being drawn through them to estimate the magnitude of passive deposition during the period of time that filter packs are exposed in the sampler (typically 24 to 72 hours). Depending on the sampling environment and passive deposition period, field blank mass concentrations range from 5 to 30 μg per 47 mm Teflon-membrane filter, and field blank organic carbon concentrations range from 20 to 40 μg per 47 mm quartz-fiber filter. As average field blank measurements exceed their associated uncertainties (e.g., standard deviations or root mean squared errors), these field blanks measurements should be subtracted from the ambient measurements to obtain realistic ambient mass and chemical concentrations.

(a) Summer 1990



(b) Winter 1995

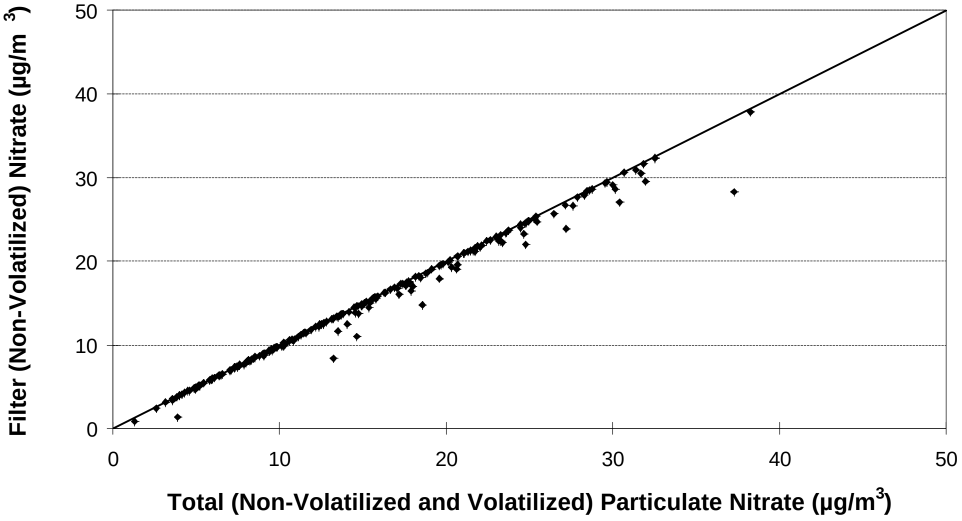


Figure 5-1. Comparison of $PM_{2.5}$ filter nitrate (non-volatilized nitrate) and total particulate nitrate (non-volatilized nitrate plus volatilized nitrate) at California's San Joaquin Valley during: (a) summer 1990, and (b) winter 1995.

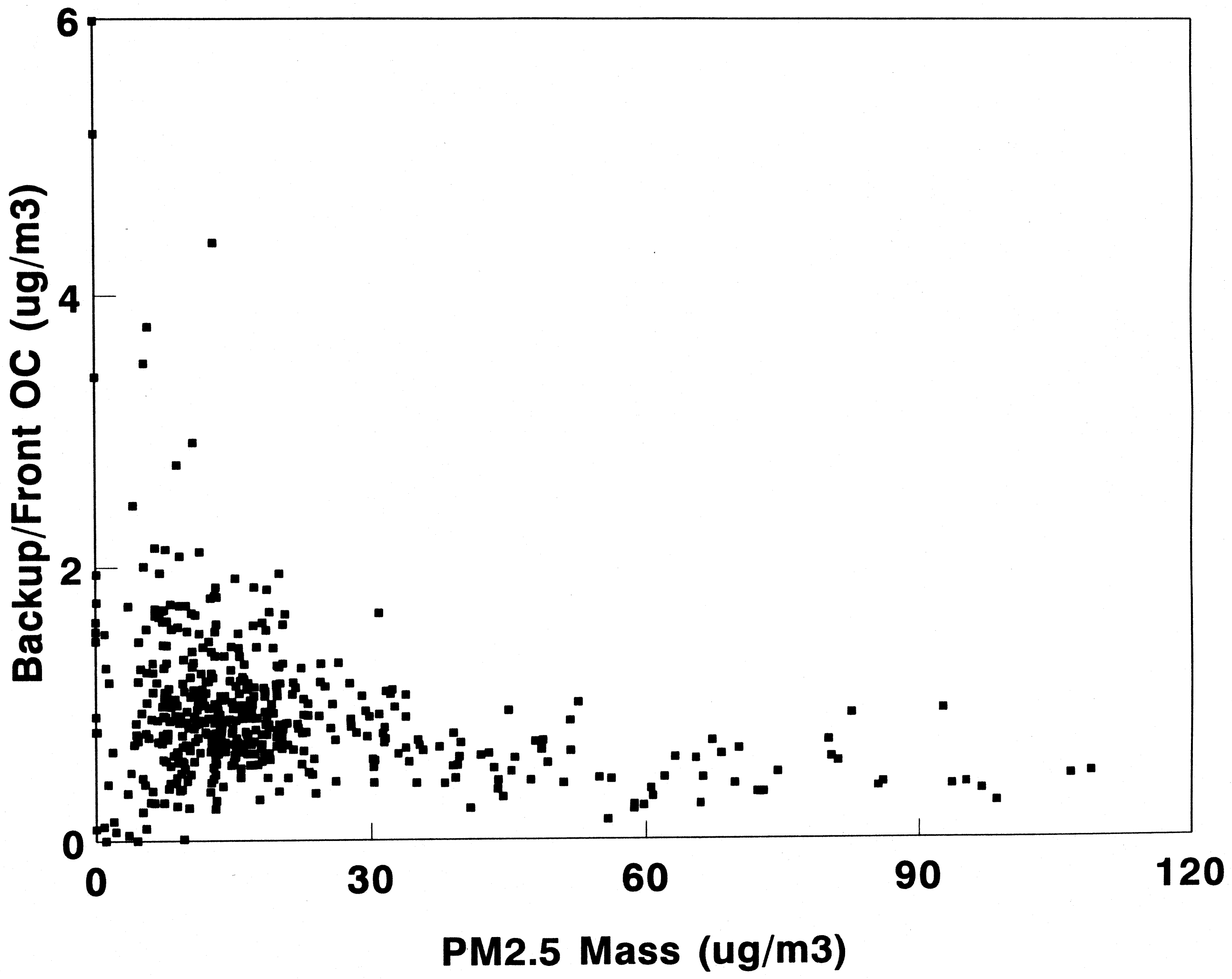


Figure 5-2. Relationship between the ratio of backup to front organic carbon and $PM_{2.5}$ mass taken at California's San Joaquin Valley during the summer of 1990.

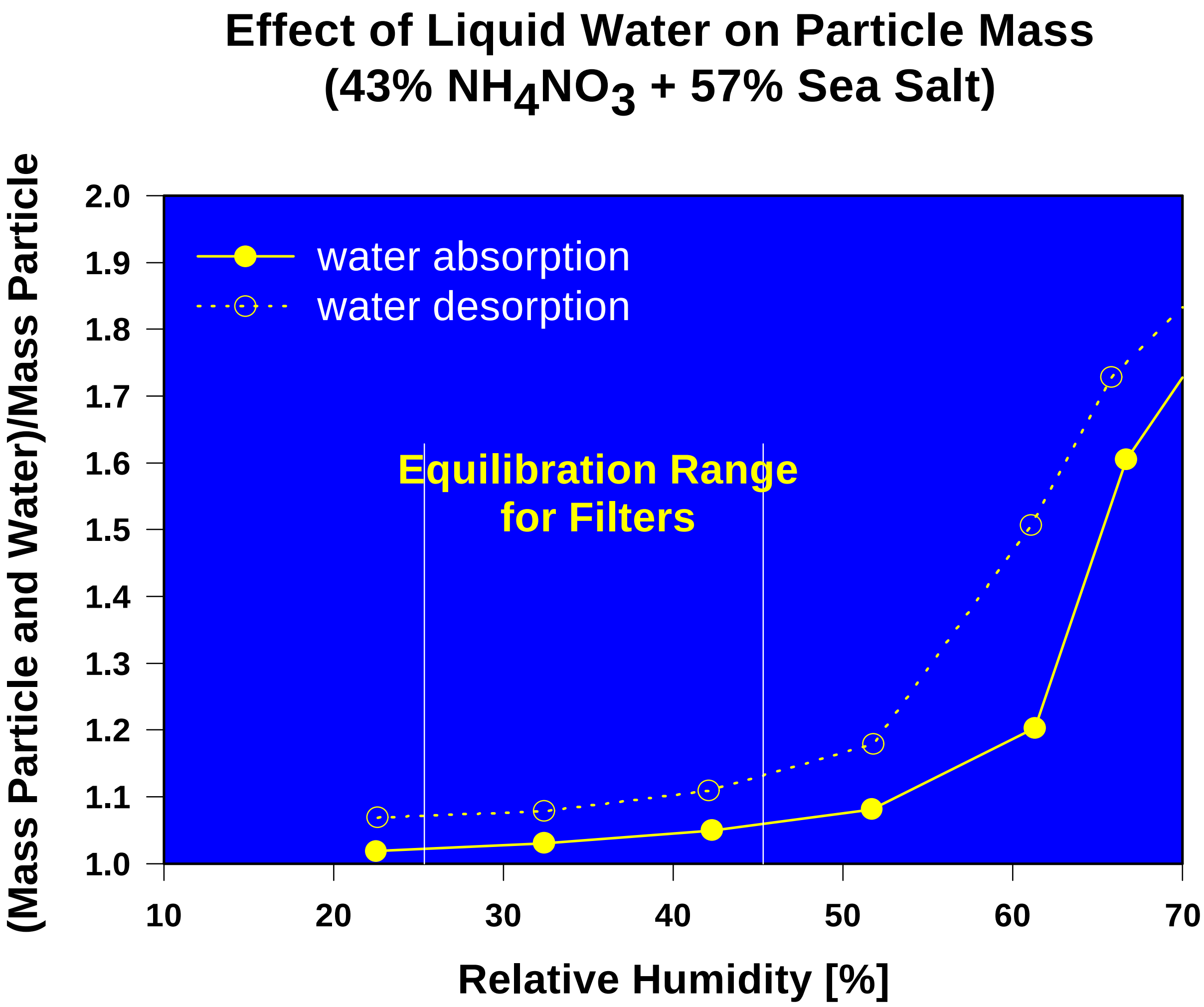


Figure 5-3. An example of the effects of liquid water on particle mass within U.S. EPA $PM_{2.5}$ filter weighing equilibration range.

6.0 QUALITY ASSURANCE

Every measurement consists of a value, a precision, an accuracy, and a validity. Quality control (QC) and quality auditing establish the precision, accuracy, and validity of measured values. Quality assurance integrates quality control and quality auditing to determine these four attributes of each environmental measurement (Watson et al., 1995b).

Quality assurance (QA) integrates quality control, quality auditing, measurement method validation, and sample validation into the measurement process. The results of quality assurance are data values with specified precisions, accuracies, and validities. Quality auditing is performed by personnel who are independent of those performing the procedures. A separate quality assurance officer performs these audits.

Quality control (QC) is the responsibility of each operator. QC is intended to prevent, identify, correct, and define the consequences of difficulties which might affect the precision and accuracy, and or validity of the measurements. The QC activities include: (1) modifying standard operating procedures (SOPs) to be followed during sampling, chemical analysis, and data processing; (2) equipment overhaul, repair, acceptance testing, and spare parts; (3) operator training, supervision, and support; (4) periodic calibrations and performance tests which include blank and replicate analyses; and (5) quality auditing.

The following section describes the requirements for SOPs and gives examples on quality auditing. Detailed quality assurance activities and requirements should follow U.S. EPA's Quality Assurance Handbook.

6.1 Standard Operating Procedures

Standard Operating Procedures (SOPs) codify the actions which are taken to implement a measurement process over a specified time period. State-of-the-art scientific information is incorporated into the SOP with each revision. Each SOP should include the following basic elements:

- A brief summary of the measurement method, its principles of operation, its expected accuracy and precision, and the assumptions which must be met for it to be valid.
- A list of materials, equipment, reagents, and suppliers. Specifications are given for each expendable item.
- Designation of the individual to be responsible for each part of the procedure.
- A general traceability path, the designation of primary standards or reference materials, tolerances for transfer standards, and a schedule for transfer standard verification.
- Start-up, routine, and shut-down operating procedures and an abbreviated checklist.

- Copies of data forms with examples of filled out forms.
- Routine maintenance schedules, maintenance procedures, and troubleshooting tips.
- Internal calibration and performance testing procedures and schedules.
- External performance auditing schedules.
- References to relevant literature and related standard operating procedures.

A Quality Assurance Handbook will be issued by U.S. EPA to address $PM_{2.5}$ monitoring with designated FRM or Class I FEMs. In addition to the field operations handbook, additional handbooks containing relevant procedures for laboratory operations and data processing/data validation operations should also be assembled for the $PM_{2.5}$ monitoring network. Table 6-1 gives an example of the SOPs required for $PM_{2.5}$ chemical speciation. These SOPs should be reviewed annually to ensure that procedures specified in the SOPs are actually being followed in field and laboratory operations.

6.2 Quality Audit Objectives

The quality auditing function consists of systems and performance audits. Systems audits start with a review of the operational and QC procedures to assess whether they are adequate to assure valid data that meet the specified levels of accuracy and precision. After reviewing the procedures, the auditor examines all phases of the measurement or data processing activity to determine that the procedures are being followed and the operational staff are properly trained. The systems audit is intended to be a cooperative assessment resulting in improved data, rather than a judgmental activity.

Performance audits establish whether the predetermined specifications are being achieved in practice. The performance audit challenges the measurement/analysis system with known standards traceable to a primary standard. For data processing, the performance audit consists of independently processing sections of the data and comparing the results. Performance objectives should be specified for the field or laboratory instruments on which performance audits are conducted. Audit findings are compared against these values to decide whether or not remedial action is needed.

6.3 Laboratory Performance Audit

The laboratory performance audit consists of the submission of known standards to routine laboratory procedures and of an interlaboratory comparison of those standards. Gravimetric analysis can be audited by weighing independent Class M or NIST-traceable standard weights and Teflon-membrane filters which will be pre-weighed and post-weighed at the primary laboratory and the audit laboratory for comparison. To audit the analysis of soluble species on quartz-fiber filters, a solution containing chloride, sulfate, sodium, nitrate, ammonium, and potassium is prepared and deposited in known amounts on quartz-fiber filters. Samples at a minimum of three concentrations are submitted to the routine chemical analyses for chloride, nitrate, and sulfate by ion chromatography, for water-soluble potassium by atomic absorption spectrophotometry, and for ammonium by automated colorimetry. To audit the analysis of the elements by x-ray fluorescence (XRF) on Teflon-membrane filters, several thin film Micromatter pure-element deposits are submitted for routine XRF analysis.

At the present time there are no widely accepted standards for elemental and organic carbon. Potassium acid phthalate (KHP) solutions can be deposited on quartz-fiber filters to create organic carbon standards. A minimum of three sets of analyses at each concentration level and three blank filters should be analyzed for each audited chemical species.

An example of field and laboratory performance audit observables is shown in Table 6-2. Since performance audits for field operations will be addressed in U.S. EPA's quality assurance handbook, only laboratory performance audits are discussed here. The quality audit function needs to be incorporated into the $PM_{2.5}$ chemical speciation monitoring network to ensure the accuracy, precision, and validity of mass and chemical measurements.

Table 6-1
Examples of Standard Operating Procedures to be Applied in the $PM_{2.5}$ Chemical Speciation Monitoring Network

Subject	Observable/Method	DRI Standard Operating Procedure
Chain-of-Custody	Filter Pack Handling	Filter Pack Assembling, Disassembling, and Cleaning Procedure
	Shipping and Receiving	Sample Shipping, Receiving, and Chain-of-Custody
Sample Pretreatment	Potassium Carbonate Impregnation Citric Acid Impregnation	Impregnating, Drying, and Acceptance Testing of Filters for Sampling Gases in Air
	Nylon Filter Cleaning	Preparation of Nylon Filters for Nitric Acid or Total Nitrate Sampling
	Quartz Filter Pre-Firing	Pre-firing of Quartz Fiber Filters for Carbonaceous Material Sampling

Table 6-1 (continued)
Examples of Standard Operating Procedures to be Applied in the $PM_{2.5}$ Chemical Speciation Monitoring Network

Subject	Observable/Method	DRI Standard Operating Procedure
Chemical Analysis	Sample Sectioning	Sectioning of Teflon and Quartz Filter Samples
	Filter Extraction	Extraction of Ionic Species from Filter Samples
	Mass	Gravimetric Analysis Procedures
	40 Elements (Na to U)	X-ray Fluorescence (XRF) Analysis of Aerosol Filter Samples
	Fluoride (F^-) Chloride (Cl^-) Nitrate (NO_3^-) Sulfate ($SO_4^=$) Sulfur Dioxide (SO_2) as $SO_4^=$	Analysis of Filter Extracts and Precipitation Samples by Ion Chromatography
	Ammonium (NH_4^+) Ammonia (NH_3) as NH_4^+	Analysis of Filter Extracts and Precipitation Samples for Ammonium by Automated Colorimetric Analysis

Table 6-1 (continued)
Examples of Standard Operating Procedures to be Applied in the $PM_{2.5}$ Chemical Speciation Monitoring Network

Subject	Observable/Method	DRI Standard Operating Procedure
	Soluble Sodium (Na^+) Soluble Potassium (K^+)	Analysis of Filter Extracts and Precipitation Samples by Atomic Absorption Spectroscopy
	High Temperature Organic Carbon (OH) Total Organic Carbon (OC) High Temperature Elemental Carbon (EH) Total Elemental Carbon (EC) Total Carbon (TC)	Thermal/Optical Reflectance Carbon Analysis of Aerosol Filter Samples
Aerosol/Gas Data	Data Validation	Dry Deposition Field, Mass, and Chemical Data Processing and Data Validation

Table 6-2
Examples of Laboratory Performance Audit Observables

Observable	Measurement Device	Performance Test Frequency	Performance Standard	Calibration Frequency	Calibration Standard	Audit Frequency	Audit Standard	Primary Standard
Aerosol								
Sample Flow	ERT/DRI Modified Sequential Filter Sampler with Bendix 240 Cyclone and HNO_3 Denuder and with SA254 PM_{10} Inlet	Once/day	Calibrated Rotameter	At the Beginning and End of one-month Sampling Period or When Performance Tests Out of Spec	Calibrated Rotameter	Once/2-months	Mass Flow Meter	Certified Roots Meter
$PM_{2.5}$ and PM_{10} Mass	Cahn 31 Electromicro-balance	1/10 Samples	NBS Class M Standard Weights	At Beginning of Weighing Session	NBS Class M Standard Weights	Once/2-months	NBS Class M Standard Weights	NBS Class M Standard Weights
		3/10 Samples	Replicate					
$PM_{2.5}$ and PM_{10} Elements	Kevex 700/8000 XRF Analyzer	1/15 Samples	NBS Thin Film Standards	Quarterly	Micromatter Thin Film Standards	Once/2-months	Prepared Standard Deposit	Thin Film Standard
		1/15 Samples	Replicate					
$PM_{2.5}$ and PM_{10} Ions (SO_4 & NO_3), and HNO_3 (g)	Dionex 2020i Ion Chromatographic Analyzer	1/10 Samples	Solution Standards	At Beginning of Each Run	ACS Certified Standard Solutions	Once/2-months	N/A	ACS Certified Chemicals
		1/10 Samples	Replicate					
Soluble $PM_{2.5}$ and PM_{10} Sodium (Na^+) Ion	Perkin-Elmer Model 2380 Atomic Absorption	1/10 Samples	Solution Standards	At Beginning of Each Run	ACS Certified Standard Solutions	Once/2-months	N/A	ACS Certified Chemicals
		1/10 Samples	Replicate					
Soluble $PM_{2.5}$ Potassium (K^+) Ion	Perkin-Elmer Model 2380 Atomic Absorption	1/10 Samples	Solution Standards	At Beginning of Each Run	ACS Certified Standard Solutions	Once/2-months	N/A	ACS Certified Chemicals
		1/10 Samples	Replicate					
$PM_{2.5}$ Ammonium and Absorbent NH_3 (g).	Technicon TRAACS 800 Colorimetric Analyzer	1/10 Samples	Solution Standards	At Beginning of Each Run	ACS Certified Standard Solutions	Once/2-months	N/A	ACS Certified Chemicals
		1/10 Samples	Replicate					
$PM_{2.5}$ Carbon	DRI/OGC Thermal/Optical Carbon Analyzer	1/10 Samples	Methane Gas	Once/2-months or When Performance Test Tolerances Not Met	Methane, CO_2 Gas and ACS Certified KHP	Once/2-months	Standard KHP Solutions	ACS Certified Chemicals
		1/10 Samples	Replicate					

7.0 DATA BASE MANAGEMENT AND DATA VALIDATION

Watson et al. (1995a) describe a measurement as having four attributes: (1) a value; (2) a precision; (3) an accuracy; and (4) a validity. Watson et al. (1995a) propose a formalism that includes standard operating procedures to ensure validity, replicate and blank analyses to estimate precision, and periodic performance audits with independent standards to determine accuracy. These need to be integral parts of all methods used to monitor compliance (U.S. EPA, 1989).

Chow et al. (1994c) defines the components of precision, accuracy, and validity of ambient aerosol measurements as follows:

- Measurement is an observation at a specific time and place which possesses four attributes: (1) value – the center of the measurement interval; (2) precision – the width of the measurement interval; (3) accuracy – the difference between measured and reference values; and (4) validity – the compliance with assumptions made in the measurement method.

- Measurement method is the combination of equipment, reagents, and procedures which provide the value of a measurement. The full description of the measurement method requires substantial documentation. Two methods may use the same sampling systems and the same analysis systems, but yield different results. This was amply demonstrated in the SCAQS pilot study to evaluate measurement methods for nitrogenous species (Hering et al., 1989). For example, two sample protocols for measuring nitric acid on nylon filters are not identical methods if one laboratory performs acceptance testing on filter media and the other does not. Blank nylon filters can absorb nitric acid when exposed to air, and need to be washed and verified before sampling. Some laboratories do this, and others do not. What appear to be minor differences between methods can result in major differences between measurement values.

- Measurement method validity is the identification of measurement method assumptions, the quantification of effects of deviations from those assumptions, the determination that deviations are within reasonable tolerances for the specific application, and the creation of procedures to quantify and minimize those deviations during that application. For the $PM_{2.5}$ speciation network, substantial effort should be placed on establishing the validity of measurement methods.

- Sample validation is accomplished by procedures which identify deviations from measurement assumptions and assign flags to individual measurements for potential deviations from assumptions. Additional validation is accomplished by comparing values with known physical relationships. For example, $PM_{2.5}$ concentrations must never exceed corresponding PM_{10} concentrations. When they do, beyond a few precision intervals, either the PM_{10} or $PM_{2.5}$ measurement is invalid.

- The comparability and equivalence of sampling and analysis methods are established by the comparison of values and precisions for the same measurement obtained by different measurement methods. Collocated sampling, interlaboratory, and intralaboratory comparisons are usually made to establish this comparability. Simultaneous measurements of the same observable are considered equivalent when more than 90% of the values differ by no more than the sum of two-sigma precision intervals for each measurement. Up to 25% of the $PM_{2.5}$ core sites should be collocated with additional FRMs to evaluate equivalence of $PM_{2.5}$ measurements.

- Completeness measures how many environmental measurements with specified values, precisions, accuracies, and validities were obtained out of the total number attainable. It measures the practicality of applying the selected measurement processes throughout the measurement period. Data bases which have excellent precision, accuracy, and validity may be of little utility if they contain so many missing values that data interpretation is impossible.

This section specifies the data base requirements, defines different levels of data validation, and discusses the requirements for substrate data processing.

7.1 Data Base Requirements

Aerosol data processing consists of six general tasks:

- **Recording.** The relevant information obtained at the time an operation is performed is registered on a data sheet, data logger, or other transfer medium.
- **Input.** The data are transferred from the recording medium into computer-accessible files.
- **Merging.** Data from various files pertaining to an individual sample or sampling day are retrieved and related to each other.
- **Calculations.** Data items are combined in mathematical expressions to yield a desired result. These include pollutant concentrations, accuracies, and precisions.
- **Data Validation.** Data are verified against earlier or redundant recordings, with calibration and operating records, and with each other.
- **Output.** Data are arranged into desired formats for input to data interpretation and modeling software.

The data base management system needs to fulfill the following requirements:

- Quantitative and descriptive information must be accommodated.
- Data from a number of sources must be merged in an efficient and cost-effective manner.
- Input data required by models should be easily accessible directly from the data base.

7.2 Data Validation

Figure 7-1 shows the general process of data validation. Data validation is the most important function of data processing. Sample validation consists of procedures which identify deviations from measurement assumptions and procedures. Three levels (Mueller et al., 1983) of validation are applied which will result in the assignment of a rating to each measurement: (1) valid; (2) valid but suspect; or (3) invalid.

Level I sample validation takes place in the field or in the laboratory and consists of the following: (1) flagging samples when significant deviations from measurement assumptions have occurred; (2) verifying computer file entries against data sheets; (3) eliminating values for measurements which are known to be invalid because of instrument malfunctions; (4) replacement of data from a backup data acquisition system in the event of failure of the primary system; and (5) adjustment of measurement values of quantifiable calibration or interference biases.

Level II sample validation takes place after data from various measurement methods have been assembled in the master data base. Level II applies consistency tests based on known physical relationships between variables to the assembled data. Chow et al. (1994) illustrate several internal consistency checks that can be applied to evaluate validity when different particle size fractions are measured and submitted to chemical analyses. These include: (1) comparisons between mass and chemical concentrations in different size fractions (e.g., $PM_{2.5}$ concentrations must always be less than or equal to PM_{10} concentrations); (2) comparisons between mass concentrations and the weighted sum of chemical species; (3) charge balances between anions and cations; and (4) comparisons between concentrations of the same species measured by different analysis methods (e.g., sulfate and chloride by IC and total sulfur and chlorine by XRF or PIXE, soluble sodium and potassium by AAS and total sodium and potassium by XRF or PIXE). Data adjustments for quantifiable biases (e.g., large particle absorption corrections for aluminum) can be made in Level II validation if they are discovered after assembly of the master data base.

Level III sample validation is part of the data interpretation process. The first assumption upon finding a measurement inconsistent with physical expectations is that the unusual value is due to a measurement error. If, upon tracing the path of the measurement, nothing unusual is found, the value can be assumed to be a valid result of an environmental cause. Unusual values are identified during the data interpretation process as the following: (1) extreme values; (2) values which would normally track the values of other variables in a time series; and (3) values for observables which would normally follow a qualitatively predictable spatial or temporal pattern.

Data validation actions at each level are recorded in a data validation summary which accompanies the data base. Data base records contain flags to identify the level of validation which they have received at any point in their existence.

7.3 Substrate Data Processing

Aerosol data processing and validation requires the following: (1) assignment of ID codes to substrates; (2) field data recording of the IDs and their corresponding sampling sites, sampling dates, sampling times, sampling durations, sample flow rates, and deviations from normal sampling procedures; (3) laboratory instrument recording of analytical outputs; (4) Level I data validation, flagging, and editing of these individual data files; (5) merging field and laboratory data for sample sets; (6) Level II data validation, editing, flagging, and re-analysis; (7) calculation of ambient concentrations and precisions; and (8) formatting and reporting of concentrations, precisions, and data validation activities. An example of a data base management system which performs these functions is illustrated in Figure 7-1.

Field data are entered into computerized data forms. Substrate IDs can be barcoded and then entered with a scanner rather than being typed. The screen forms have limits that do not allow entry of values lying outside of a certain range. Every data item entered is verified by the data processing supervisor against the original data sheet.

A data base structure which contains fields for chemical concentrations and their uncertainties is formed. Each record contains sample IDs, sample volumes, sample times, sampling sites, and sampling dates are integrated into this structure from the field file. All other fields contain the missing data default value. These defaults are replaced by laboratory analysis data as they become available. In this way, it is always possible to determine which analyses have been completed and which have not.

The laboratory chain-of-custody data base records the disposition of each sample and this data base can be consulted to determine the fate of missing values in the master data base. This independent tracking is needed to prevent sample IDs from being mixed up.

Every laboratory analysis instrument should be linked to IBM-PC compatible computers, and data should be recorded in Xbase (*.DBF) or ASCII text files. Barcode readers can be used to enter each analysis ID for an analysis run. Samples are keyed to sample ID codes, and data base programs associate records in the laboratory files with data in the master file. These programs also replace the defaults in the master data file with the laboratory values. Separate flags are entered at the time of analysis to indicate if a sample is an ambient sample, a source sample, a field blank, a laboratory blank, a replicate, a re-run, a performance test standard, or an audit standard. These flags are used to separate these quality control values from the individual data bases to generate quality control charts and precision estimates. Examples of field and laboratory validation flags are shown in Tables 7-1 and 7-2.

When data for a record have been assembled, Level II data validation comparisons should be conducted, as discussed in Section 7.2. These additional validity checks should be applied to chemically-speciated particle samples whenever possible. Statistical summaries, scatterplots, and time series plots of selected species concentrations are produced to identify outliers for investigation and potential re-run. A data validation summary is maintained in the character field associated with each record to provide a traceability trail for data adjustments, replacements, or deletions.

When sample concentration data have been assembled, the data base program creates another data base of ambient concentrations. Propagated precision and blank subtraction calculations are made at this stage. The field and laboratory data validation flags are assigned as part of the data validation process. Data validation summaries accompany this final data base.

Table 7-1
Examples of Ambient Field Sampling Data Validation Flags[a]

Validation Flag	Sub Flag	Description
A		Sampler adjustment or maintenance.
	A1	Sampler audit during sample period.
	A2	Sampler cleaned prior to sample period.
	A3	Particle size cut device regreased or replaced prior to sample period.
B		Field Blank.
D		Sample dropped.
	D1	Sample dropped after sampling.
	D2	Filter dropped during unloading.
F		Filter damaged or ripped.
	F1	Filter damaged in the field.
	F2	Filter damaged when removed from holder.
	F3	Filter wrinkled.
	F4	Filter torn due to over-tightened filter holder.
	F5	Teflon membrane separated from support ring.
	F6	Pinholes in filter.
G		Filter deposit damaged.
	G1	Deposit scratched or scraped, causing a thin line in the deposit.
	G2	Deposit smudged, causing a large area of deposit to be displaced.
	G3	Filter returned to lab with deposit side down in PetriSlide.
	G4	Part of deposit appears to have fallen off; particles on inside of PetriSlide.
	G5	Finger touched filter in the field (without gloves).
	G6	Finger touched filter in the lab (with gloves).
H		Filter holder assembly problem.
	H1	Filter misaligned in holder - possible air leak.
	H2	Filter holder loose in sampler - possible air leak.
	H3	Filter holder not tightened sufficiently - possible air leak.
	H4	Filter support grid upside down.
	H5	Two substrates loaded in place of one.
I		Inhomogeneous sample deposit.
	I1	Evidence of impaction - deposit heavier in center of filter.
	I2	Random areas of darker or lighter deposit on filter.
	I3	Light colored deposit with dark specks.
	I4	Non-uniform deposit near edge - possible air leak.
L		Sample loading error.
	L1	Teflon and quartz filters were loaded reversely in SFS.
	L2	$PM_{2.5}$ and PM_{10} filter pack switched.
	L3	Fine and Coarse filters were loaded reversely in dichotomous sampler.
	L4	Filter loaded in wrong port.

Table 7-1 (continued)
Examples of Ambient Field Sampling Data Validation Flags[a]

Validation Flag	Sub Flag	Description
M		Sampler malfunction.
N		Foreign substance on sample.
	N1	Insects on deposit, removed before analysis.
	N2	Insects on deposit, not all removed.
	N3	Metallic particles observed on deposit.
	N4	Many particles on deposit much larger than cut point of inlet.
	N5	Fibers or fuzz on filter.
	N6	Oily-looking droplets on filter.
	N7	Shiny substance on filter.
	N8	Particles on back of filter.
	N9	Discoloration on deposit.
O		Sampler operation error.
	O1	Pump was not switched on after changing samples.
	O2	Timer set incorrectly.
	O3	Dichotomous sampler assembled with virtual impactor 180° out of phase; only PM_{10} data reported.
P		Power failure during sampling.
Q		Flow rate error.
	Q1	Initial or final flow rate differed from nominal by > ±10%.
	Q2	Initial or final flow rate differed from nominal by > ±15%.
	Q3	Final flow rate differed from initial by > ±15%.
	Q4	Initial or final flow rate not recorded, used estimated flow rate.
	Q5	Nominal flow rate assumed.
R		Replacement filter used.
	R1	Filter that failed flow rate or QC checks replaced with spare.
	R2	Filter sampling sequence changed from order designated on field data sheet.
S		Sample validity is suspect.
T		Sampling time error.
	T1	Sampling duration error of > ±10%.
	T2	Sample start time error of > ±10% of sample duration.
	T3	Elapsed time meter reading not recorded or recorded incorrectly. Sample duration estimated based on readings from previous or subsequent sample.
	T4	Nominal sample duration assumed.
	T5	Sample ran during prescribed period, plus part of next period.
	T6	More than one sample was run to account for the prescribed period.

Table 7-1 (continued)
Examples of Ambient Field Sampling Data Validation Flags[a]

Validation Flag	Sub Flag	Description
U		Unusual local particulate sources during sample period.
	U1	Local construction activity.
	U2	Forest fire or slash or field burning.
V		Invalid sample (Void).
W		Wet Sample.
	W1	Deposit spotted from water drops.
	W2	Filter damp when unloaded.
	W3	Filter holder contained water when unloaded.
X		No sample was taken this period, sample run was skipped.

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[a] Samples are categorized as valid, suspect, or invalid. Unflagged samples, or samples with any flag except 'S' or 'V' indicate valid results. The 'S' flag indicates samples of suspect validity. The 'V' flag indicates invalid samples. Field data validation flags are all upper case.

Table 7-2
Examples of Chemical Analysis Data Validation Flags[a]

Validation Flag	Sub Flag	Description
b		Blank.
	b1	Field/dynamic blank.
	b2	Laboratory blank.
	b3	Distilled-deionized water blank.
	b4	Method blank.
	b5	Extract/solution blank.
	b6	Transport blank.
c		Analysis result reprocessed or recalculated.
	c1	XRF spectrum reprocessed using manually adjusted background.
d		Sample dropped.
f		Filter damaged or ripped.
	f1	Filter damaged, outside of analysis area.
	f2	Filter damaged, within analysis area.
	f3	Filter wrinkled.
	f4	Filter stuck to PetriSlide.
	f5	Teflon membrane separated from support ring.
	f6	Pinholes in filter.
g		Filter deposit damaged.
	g1	Deposit scratched or scraped, causing a thin line in the deposit.
	g2	Deposit smudged, causing a large area of deposit to be displaced.
	g3	Filter deposit side down in PetriSlide.
	g4	Part of deposit appears to have fallen off; particles on inside of PetriSlide.
	g5	Ungloved finger touched filter.
	g6	Gloved finger touched filter.
h		Filter holder assembly problem.
	h1	Deposit not centered.
	h2	Sampled on wrong side of filter.
	h4	Filter support grid upside down- deposit has widely spaced stripes or grid pattern.
	h5	Two filters in PetriSlide- analyzed separately.
i		Inhomogeneous sample deposit.
	i1	Evidence of impaction - deposit heavier in center of filter.
	i2	Random areas of darker or lighter deposit on filter.
	i3	Light colored deposit with dark specks.
	i4	Non-uniform deposit near edge - possible air leak.

Table 7-2 (continued)
Examples of Chemical Analysis Data Validation Flags[a]

Validation Flag	Sub Flag	Description
m		Analysis results affected by matrix effect.
	m1	Organic/elemental carbon split undetermined due to an apparent color change of non-carbon particles during analysis; all measured carbon reported as organic.
	m2	Non-white carbon punch after carbon analysis, indicative of mineral particles in deposit.
	m3	A non-typical, but valid, laser response was observed during TOR analysis. This phenomena may result in increased uncertainty of the organic/elemental carbon split. Total carbon measurements are likely unaffected.
n		Foreign substance on sample.
	n1	Insects on deposit, removed before analysis.
	n2	Insects on deposit, not all removed.
	n3	Metallic particles observed on deposit.
	n4	Many particles on deposit much larger than cut point of inlet.
	n5	Fibers or fuzz on filter.
	n6	Oily-looking droplets on filter.
	n7	Shiny substance on filter.
	n8	Particles on back of filter.
	n9	Discoloration on deposit.
q		Standard.
	q1	Quality control standard.
	q2	Externally prepared quality control standard.
	q3	Second type of externally prepared quality control standard.
	q4	Calibration standard.
r		Replicate analysis.
	r1	First replicate analysis on the same analyzer.
	r2	Second replicate analysis on the same analyzer.
	r3	Third replicate analysis on the same analyzer.
	r4	Sample re-analysis.
	r5	Replicate on different analyzer.
	r6	Sample re-extraction and re-analysis.
	r7	Sample re-analyzed with same result, original value used.
s		Suspect analysis result.
v		Invalid (void) analysis result.
	v1	Quality control standard check exceeded ± 10% of specified concentration range.
	v2	Replicate analysis failed acceptable limit specified in SOP.
	v3	Potential contamination.
	v4	Concentration out of expected range.

Table 7-2 (continued)
Examples of Chemical Analysis Data Validation Flags[a]

Validation Flag	Sub Flag	Description
w		Wet Sample.
	w1	Deposit spotted from water drops.

---

[a] Analysis results are categorized as valid, suspect, or invalid. Unflagged samples, or samples with any flag except 's' or 'v' indicate valid results. The 's' flag indicates results of suspect validity. The 'v' flag indicates invalid analysis results. Chemical analysis data validation flags are all lower case.

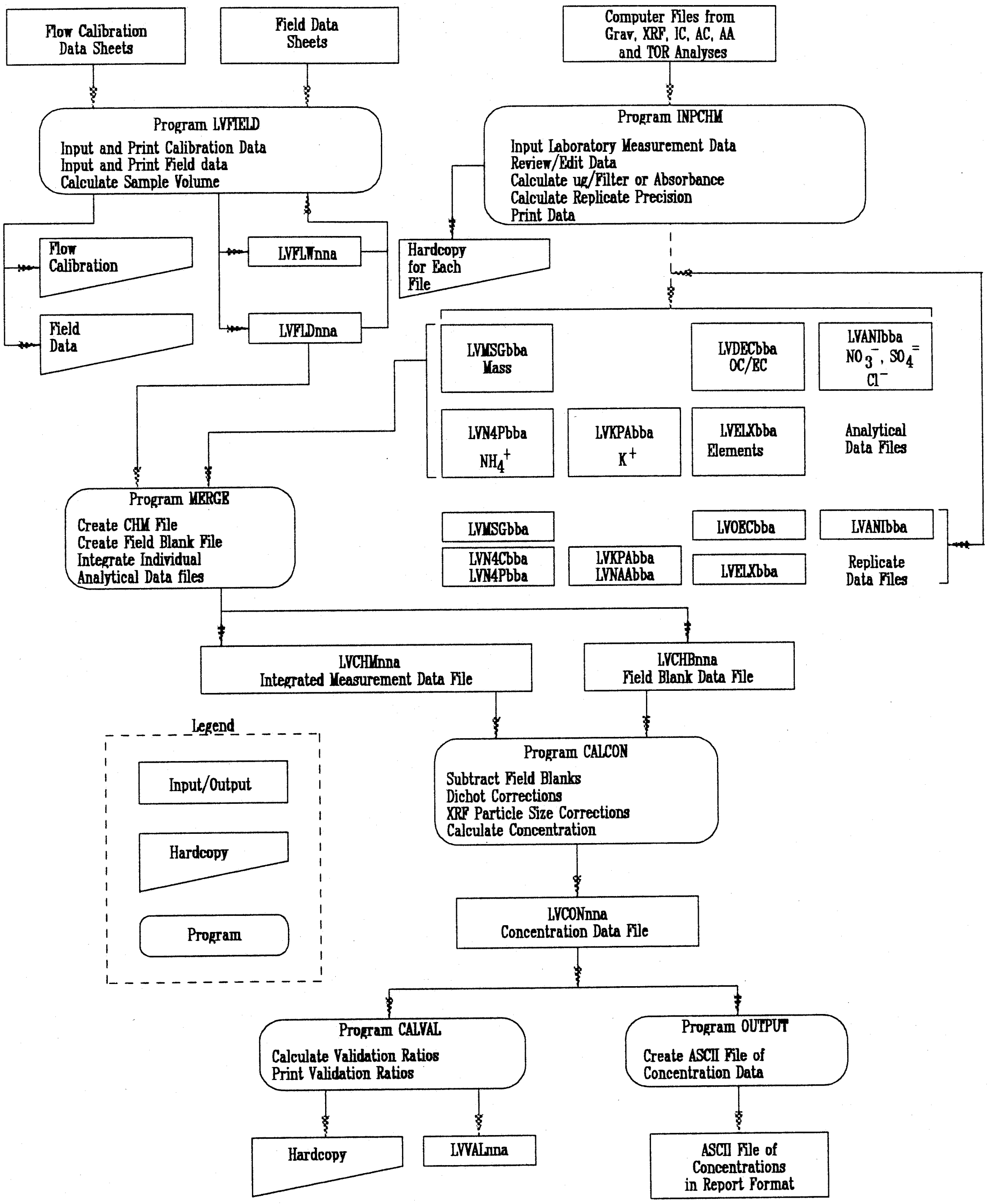

Figure 7-1. Example of an aerosol data processing and validation system.

8.0 MONITORING STRATEGIES

The preceding sections identified different methods for PM sampling and chemical analysis. These sections showed that chemical analysis of $PM_{2.5}$ or PM_{10} samples must be closely coupled with the appropriate sampling methods and filter handling procedures. This section organizes that general knowledge into specific steps that can be applied when planning a $PM_{2.5}$ measurement study (Chow and Watson, 1994a). It also emphasizes the importance of field/laboratory integration. The analytical laboratory should be involved at the sampler design stage to assure compatibility among sampling methods, analysis methods, filter media, and lower quantifiable limits.

8.1 General Approach

The first step is to determine the specific monitoring objectives. Compliance determination, health effects evaluation, source apportionment, and control strategy assessment are the most common objectives for $PM_{2.5}$ monitoring. Data from $PM_{2.5}$ speciation monitoring networks may need to be augmented by additional sampling for transition metals, organics, and single particle characterizations. Compliance $PM_{2.5}$ monitoring networks do not provide samples amenable to all chemical analyses because of the limitations of single-filter media. Source apportionment and control strategy evaluation require chemical speciation, so additional measures must be taken when these objectives are to be addressed.

The second step is to determine which chemicals need to be measured and at what levels they are expected. When source apportionment is an objective, it is desirable to obtain chemicals which are present in the sources which are suspected of contributing to $PM_{2.5}$.

The potential contributors can often be determined from emissions inventory summaries in the study area. These inventories should include emissions estimates for suspended particles, carbon monoxide (CO), sulfur dioxide (SO_2), nitrogen oxides (NO_x), volatile organic compounds (VOCs), and ammonia (NH_3), if possible. The gaseous precursors are needed to assess whether or not secondary aerosol might contribute to elevated $PM_{2.5}$ concentrations.

When gridded inventories are available, or the locations of point sources are known, these should be examined to determine the locations of emitters relative to sampling locations. The closer the source, the greater the probability that some of the chemicals it emits will be detected at the receptor. Emissions events should be identified, such as prescribed fires, wildfires, construction and demolition activities, and plant upsets.

These may have a different chemical character from the sources which are listed in the inventory. Agricultural extension offices should be contacted to obtain land-use maps and soil conservation surveys. Periods of tilling, fertilizing, and grazing might be indicative of elevated emissions from these activities. Local fire departments, the National Forest Service, the Bureau of Land Management, and other fire management agencies can often supply information on local burning events.

Microinventories are also helpful for identifying potential contributors and the chemical species which correspond to these contributions (Pace, 1979). Microinventories include detailed surveys and locations of vacant lots, storage piles, major highways, construction sites, and industrial operations. These are plotted on a map with notes regarding the visual appearance of each potential emitter. For example, if chimneys are present in a residential neighborhood, this observation is recorded and photographs are taken. Roads in the vicinity of sampling sites are classified with respect to the type of traffic on them and whether or not they have sidewalks and paved shoulders.

Expected emissions cycles should be examined to determine sampling periods and durations. For example, residential woodburning will usually show up on samples taken during the night whereas agricultural burning will usually show up during the daytime. While these two source types may be indistinguishable based on their chemical profiles shown in Figure 2-2, their diurnal cycles will provide convincing evidence that one or the other is a major contributor when both activities occur simultaneously.

Particle size is of value in separating one source from another. Particle size fractions, chemical analyses, sampling frequencies, and sample durations need to be considered because more frequent samples, or samples taken at remote locations, may require a sequential sampling feature to minimize operator costs. Shorter sample durations may require a larger flow rate to obtain an adequate sample deposit for analysis. The types of analyses and size fractions desired affect the number of sampling ports and different filter media needed.

The third step is to calculate the expected amount of deposit on each filter for each chemical species and compare it to typical detection limits listed in Tables 4-1 and 4-2 for the analyses being considered. Urban samples acquire adequate deposits for analysis with flow rates as low as ~20 L/min for as short as 4-hour sample durations. Samples at non-urban sites may require >100 L/min flow rates for 24-hour durations to obtain an adequate deposit for chemical speciation.

The fourth step is to apply, create, adapt, or purchase the sampling system which provides the most cost-effective and reliable means of meeting the monitoring needs. Table 3-6 identifies several sampler designs which have been applied to PM studies. FRM is used to determine compliance with the $PM_{2.5}$ standards. In complicated situations, however, especially those with many contributing sources, unknown sources, or secondary contributions, more complex sampling systems are needed which do not have reference status. Both reference and research sampling systems have been operated side-by-side in many PM_{10} studies when this is the case, and the same practice can be applied to $PM_{2.5}$ monitoring.

The final step is to create a written program plan which specifies the study objectives, sampling locations, analysis methods, filter media, sampling systems, sampling frequencies and durations, nominal flow rates, methods and schedules for inlet cleaning, calibration and performance tests, filter transport and handling procedures, database management system, data analysis methods, and record keeping protocols. A representative flow diagram of sampling and analysis strategies is shown in Figure 8-1, while Table 8-1 contains a typical outline for a study plan. Such a program plan is essential for assuring that all participants know what roles they are required to carry out as part of a $PM_{2.5}$ study.

The procedure outlined above describes an ideal program which may require several hundred thousand dollars to complete. Such expenditures are often worthwhile when costly pollution control decisions must be made, since these decisions may result in tens of millions of dollars of expenditures. These expenditures cannot always be justified without some pilot studies using existing equipment and samples to provide screening analysis. Sometimes these initial analyses can provide information which is sufficient to design the desired control strategy, and further measurements are not needed. The following sub-sections provide guidance on what can be done with different sampling and analysis configurations in a step-wise fashion.

8.2 Analysis of Archived $PM_{2.5}$ FRM Filters

Current regulations (section 2.8.15 of 40 CFR part 58) require air pollution control agencies to archive $PM_{2.5}$ filters from all SLAMS sites for a minimum of one year after collection (U.S. EPA, 1997a). These filters can be made available for supplemental chemical analysis to provide additional information on $PM_{2.5}$ chemical composition. Elemental, single particle, and/or ion analyses can be applied to these filters subject to the limitations discussed in Section 4.

The archived filters of greatest interest are those which exhibit $PM_{2.5}$ concentrations in excess of 65 $\mu g/m^3$. Filters from all sites within the air quality management area on an exceedance day should be examined, even though the $PM_{2.5}$ standard may not be exceeded at every site. Differences in chemical content among sites, coupled with knowledge of emission source locations, will assist in determining whether or not chemical contributions have a local or regional effect on $PM_{2.5}$ concentrations. Field and laboratory blanks corresponding to the same lots as archived samples should also be submitted to the same chemical analyses as the exposed filters. The levels of chemical concentrations in these blanks provide information on how the chemical concentrations on the exposed filters should be interpreted.

Depending on the source mixture in the sampled area, the Teflon-membrane filters can be submitted for elemental speciation, single particle, and/or ion analysis with the following options:

- **Option I:** If trace elements, single particle characteristics, and ions are of interest, these samples can be first submitted to non-destructive analyses by XRF or PIXE to determine concentrations of ~40 trace elements between sodium (11) and uranium (92). After the elemental data are evaluated, the Teflon-membrane filters can either be (1) resuspended and re-deposited onto polycarbonate-membrane filters for single-particle analysis of particle size, morphology, and composition; or (2) extracted in DDW for ionic speciation of Cl^-, NO_3^-, $SO_4^=$, NH_4^+, Na^+, and K^+. Because of the hydrophobic nature of Teflon-membrane filters, small amounts of ethanol or other wetting agents should be added to the exposed filter surface to ensure 100% extraction efficiency. Since XRF and PIXE analyses are performed under vacuum to achieve maximum efficiency and sensitivity, some volatile compounds (such as nitrate and chlorine) evaporate during analysis, so the nitrate and chloride measurements are often underestimated.

- **Option II:** If vegetative burning or diesel exhaust is a major emission source in the sampled environment, light transmission measurements can be applied using blank filter b_{abs} as a pre-b_{abs} concentration to provide a first-order estimate of elemental carbon concentration. After light absorption analysis, the same Teflon-membrane filter can also be submitted for analysis of elements, single particles, or ions listed in Option I.

- **Option III:** If secondary inorganic aerosol such as nitrate, ammonium, and sulfate are suspected to be the major components of $PM_{2.5}$, the Teflon-membrane filter can be submitted directly to ionic speciation. This option does not allow additional speciation to be performed since the filter will be destroyed during extraction.

8.3 Variations to FRM or FEM Sampling

If it is known that chemical speciation is anticipated within the state and local monitoring network, some precautions can be taken prior to field sampling. Most importantly, acceptance testing (see Section 4.2) needs to be performed on each chemical to be quantified. If the filter cassette can be modified to accommodate multiple filters, the followig options can be taken with the existing FRMs:

- **Option I:** To address nitrate volatilization, modify the existing FRM filter cassette to add a nylon-membrane or sodium-chloride-impregnated cellulose-fiber filter behind the Teflon-membrane filter to measure total particulate nitrate (i.e., non-volatilized plus volatilized particulate nitrate). Note that a physical separation between the Teflon-membrane and impregnated filter is required to avoid cross-contamination due to the wetness of the impregnated filter. Configuring a nylon-membrane filter behind the Teflon-membrane filter will create a much larger pressure drop and may clog the flow due to high flow resistance. This configuration assumes that the anodized aluminum sampling surface in the FRM serves as an adequate nitric acid denuder to remove gaseous nitric acid. To ensure the removal of gaseous nitric acid, anodized denuders can also be installed in the FRM between the PM_{10} and WINS inlets.

- **Option II:** To address organic artifacts, modify the existing FRM filter cassette to add a quartz-fiber filter behind the Teflon-membrane filter to measure gaseous organic adsorption. A drain disc (resistance-free paper disc) can be used to physically separate the Teflon-membrane and quartz-fiber filters. Various versions of organic denuders can also be installed in the FRM between the PM_{10} and WINS inlets to estimate gaseous organic desorption.

- **Option III:** To obtain complete chemical speciation, collocate two FRMs per site. In the first unit, use a Teflon-membrane/drain disc/quartz-fiber filter pack (described in Option II) to measure mass, light absorption, elements, single particle (optional), and artifact organic carbon. If XRF or PIXE non-destructive analysis were performed, these filters can then be submitted for single particle analysis of particle size, shape, color, and additional composition. In the second unit, use a front quartz-fiber filter with backup sodium-chloride-impregnated or nylon filter pack to measure ions ($SO_4^=$, Cl^-, NH_4^+, K^+), non-volatilized and volatilized nitrate, organic and elemental carbon, organics (optional), and transition metals (optional).

In addition to module A of the IMPROVE sampler described in Section 3.3, additional modules B and C can be added to fulfill first-order chemical speciation. In the case of 24-hour sampling, continuous instruments such as Class III candidate FEMs can be collocated at the site to provide a better understanding of diurnal variations of $PM_{2.5}$ concentrations. Sequential samplers can be used to determine multi-day buildup of a variety of sources; it is desirable to have daily samples available that can be submitted to chemical speciation.

8.4 Saturation Sampling

There may be cases where one or more source categories are identified as major contributors to elevated $PM_{2.5}$, but the chemical profiles of specific emitters are too similar to differentiate them from each other. In this situation, Battery-powered Minivol $PM_{2.5}$ portable survey samplers using Teflon-membrane filters can be located within and around the suspected emitters (Watson et al., 1991b; Chow and Watson., 1997b).

If the objective of the study is to characterize fugitive dust sources, mass and elemental analyses are sufficient to separate this source category from others by receptor modeling. If ammonia is suspected to be a major source in the area, a citric-acid-impregnated filter can be placed behind the Teflon-membrane filter to address the spatial variations of $PM_{2.5}$ mass and ammonia (Chow and Egami, 1997). Several studies have applied the portable survey sampling approach to characterize the impact of residential wood combustion. In this case, collocated samplers with Teflon-membrane and quartz-fiber filters are required for full chemical speciation. The major chemical components of $PM_{2.5}$ can be used in spatial receptor models to identify the locations of specific emissions sources.

8.5 Precursor Gaseous Sampling

In cases where secondary ammonium sulfate and ammonium nitrate are major contributors, one or more sites should be operated to obtain precursor concentrations of nitric acid and ammonia gas (e.g., Chow et al., 1993c). In the eastern United States, sulfuric acid and ammonium bisulfate are also important components. In this situation, denuder methods can be applied to obtain accurate measures of the secondary aerosol and the precursor gases. These precursor gas measurements should be accompanied by collocated temperature and relative humidity measures so that equilibrium receptor models (e.g., Watson et al., 1994a) can be applied to determine whether the secondary particles are limited by ammonia or oxides of nitrogen emissions.

Table 8-1
Example of Program Plan Outline for $PM_{2.5}$ Measurement and Modeling

1.0 INTRODUCTION

- 1.1 Background
- 1.2 Objectives
- 1.3 Overview

2.0 AIR QUALITY IN THE STUDY AREA

- 2.1 Emissions
- 2.2 Meteorology
- 2.3 Atmospheric Transformations
- 2.4 Historical Air Quality Data
- 2.5 Implications for $PM_{2.5}$ Study Design

3.0 DATA ANALYSIS AND MODELING

- 3.1 Data Evaluation
- 3.2 Descriptive Air Quality Analysis
- 3.3 Descriptive Meteorological Analysis
- 3.4 Source Profile Compilation
- 3.5 Emissions Inventory
- 3.6 Receptor Model Source Apportionments
- 3.7 Trajectory Modeling
- 3.8 Secondary Aerosol Modeling
- 3.9 Case Study Descriptions

4.0 PROPOSED AMBIENT MONITORING NETWORK

- 4.1 Sampling Sites
- 4.2 Sampling Frequency and Duration
- 4.3 Sampling Methods

5.0 EMISSIONS CHARACTERIZATION

- 5.1 Emissions Activities and Microinventories
- 5.2 Geological Source Profiles
- 5.3 Motor Vehicle Exhaust Characterization
- 5.4 Residential Wood Combustion Characterization
- 5.5 Industrial Source Emission Characterization
- 5.6 Other Source Characterization

Table 8-1 (continued)
Example of Program Plan Outline for PM_{10} Measurement and Modeling

6.0 LABORATORY OPERATIONS

- 6.1 Substrate Preparation
- 6.2 Gravimetric Analysis
- 6.3 Light Absorption
- 6.4 Elemental Analysis
- 6.5 Filter Extraction
- 6.6 Ion Analysis
- 6.7 Carbon Analysis
- 6.8 Specialized Analysis

7.0 QUALITY ASSURANCE

- 7.1 Standard Operating Procedures
- 7.2 Quality Audits
- 7.3 Standard Traceability
- 7.4 Performance Tests

8.0 DATA PROCESSING, DATA BASE MANAGEMENT, AND CHAIN-OF-CUSTODY

- 8.1 Data Base Requirements
- 8.2 Levels of Data Validation
- 8.3 Continuous Data Processing
- 8.4 Substrate Data Processing

9.0 MANAGEMENT, REPORTING, AND SCHEDULE

- 9.1 Tasks and Responsibilities
- 9.2 Resource Requirements
- 9.3 Reports
- 9.4 Schedule and Milestones

10.0 REFERENCES

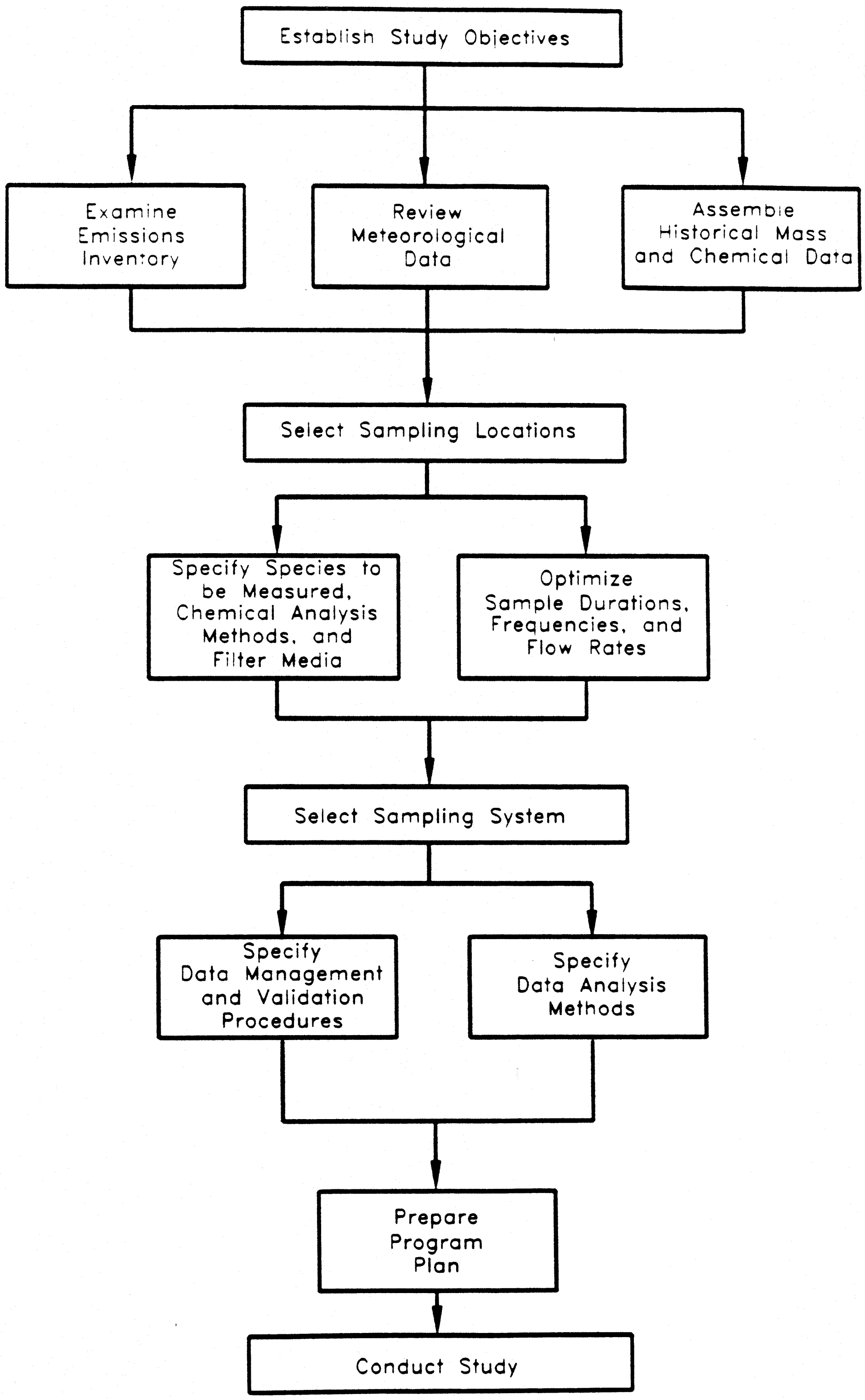

Figure 8-1. Steps in designing a speciated $PM_{2.5}$ study.

9.0 SUMMARY

Chemical characterization of suspended particles is necessary, along with the application of source apportionment models, to attribute ambient concentrations to their sources for the development of emissions reduction strategies and to establish associations between particles and health. This document identifies current technology for the sampling and analysis of $PM_{2.5}$, including FRM/FEM sampling approaches to the $PM_{2.5}$ monitoring network and chemical constituents on filter deposits.

Particle sampling on filters is the most practical method currently available to characterize the sizes and chemical compositions of PM_{10}, $PM_{2.5}$, and their sub-fractions. Ambient aerosol sampling systems consist of a combination of monitoring hardware, filter media, laboratory methods, and operating procedures which are specifically tailored to different monitoring objectives. No single sampling system can meet all needs, and it is often necessary to adapt existing sampling components to the specific situation being studied. Examples of successful sampling systems which can be copied or modified to meet these specific needs have been identified.

Chemical analysis of filter deposits **cannot** be separated from the methods used to obtain the sample. Sampling for chemical analysis requires stringent attention to choice of filter media, sample handling, sample storage, and to the sampler used to obtain the filter deposits. When chemical analysis is intended for source apportionment modeling, FRMs, FEMs, IMPROVE samplers, sequential sampling systems, particle and gas sampling systems with denuders, battery-powered samplers, dichotomous samplers, or a combination of several samplers may be needed.

An overview of filter-based particle and gas sampling systems has been provided. These systems consist of more than the mechanical device used to acquire the sample. The laboratory analyses to be applied, the type of filters which are amendable to those analyses, the minimum deposits needed on these filters, the sampling hardware which extracts pollutants from the atmosphere onto the filters, and the procedures which assure the accuracy, precision, and validity of the acquired atmospheric concentrations must all be considered.

General steps to formulate the sampling and analysis strategies have been given. A written program plan needs to be assembled which specifies the study objectives, sampling locations, analysis methods, filter media, sampling systems, sampling frequencies and durations, nominal flow rates, methods and schedules for sampler maintenance, calibration and performance tests, filter transport and handling procedures, data base management system, data analysis methods, and record keeping protocols. This plan is an evolving document, and remains in draft form until the majority of the program activities have been executed. The plan needs to be revised and finalized to reflect the actual conduct of the study and to identify improvements which should be incorporated into future plans.

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