

Fresno Supersite Final Report

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Report Prepared for: U.S. Environmental Protection Agency US EPA OAQPS (C304-02) Research Triangle Park, NC

March 31, 2005

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FRESNO SUPERSITE

FINAL REPORT

1. Introduction

The U.S. Environmental Protection Agency's (EPA) Supersites program intended to operate research-grade air monitoring stations to improve understanding of measurement technologies, source contributions and control strategies, and effects of suspended particles on health. The Fresno Supersite was one of two prototype sites established with focuses on: 1) testing and evaluation of non-routine monitoring methods, with the intent to establish their comparability with existing methods and determine their applicability to air quality planning, exposure assessment, and health impact determination; 2) increasing the knowledgebase of aerosol characteristics, behavior, and sources so regulatory agencies can develop standards and strategies that protect public health; and 3) acquiring measurements that can be used to evaluate relationships between aerosol properties, co-factors, and observed health end-points.

Specific hypotheses were formulated related to each of these objectives. Measurement methods were selected to provide the variables needed to test these hypotheses. Supersite measurements were coordinated with health and toxicological studies in the region, specifically a multi-million dollar effort by the state of California to better understand the relationships between air quality and asthma. Previous studies show that emissions, aerosol composition, and meteorology change substantially over the course of a day, between seasons, and between different years in central California where Fresno is located. It was conjectured that relationships between different measurement methods, aerosol characteristics, and health endpoints depend on these changes. Evaluating these relationships required frequent sampling over short durations for a multi-year monitoring period.

Supersite observables included *in- situ*, continuous, and short duration measurements of: 1) $PM_{2.5}$, PM_{10} , and coarse (PM_{10} minus $PM_{2.5}$) mass; 2) $PM_{2.5}$ sulfate, nitrate, carbon, light absorption, and light extinction; 3) numbers of particles in discrete size bins ranging from 0.01 to ~10 µm; 4) criteria pollutant gases (O_3 , CO, NO_x); 5) reactive gases (NO_y , NO_2 , HNO_3 , NH_3); and 6) single particle characterization by time of flight mass spectrometry. Field sampling and laboratory analysis were applied for: 1) gaseous and particulate organic compounds (light hydrocarbons, heavy hydrocarbons, carbonyls, polycyclic aromatic hydrocarbons [PAH] and other semi-volatiles); and 2) $PM_{2.5}$ mass, elements, ions, and carbon. Three satellite sites were operated next to a nearby heavily traveled roadway, in a nearby neighborhood that is influenced by wintertime wood burning, and in a non-urban area south of the Fresno city limits from 1999 to 2001. These sites were used to evaluate deviations in Supersite measurements owing to source proximity and isolation from urban emitters.

Data analysis activities were defined that related every set of measurements to a set of hypotheses that was tested. Table 1 summarizes the hypotheses, background, data analysis and method associated with each objective. These hypotheses took advantage of the long-term record of the Fresno Supersite.

Hypotheses	Background	Data Analysis and Methods and Data Used
1. Measurement Evaluation Hypotheses		
1.1 Mass and chemical (elements, ions, and carbon) measurements from routine filter samplers with a Teflon- membrane filter (from Federal Reference Method [FRM] or saturation monitors) represent actual PM _{2.5} mass within the spatial zone of representation of a CORE site.	CORE sites, such as Fresno First Street, that meet $PM_{2.5}$ siting criteria (Watson et al., 1997a), are intended to approximate the exposure of many people to $PM_{2.5}$ in outdoor air. There are few empirical measurements to verify that these criteria are adequate to accomplish this objective.	Compare mass and elemental concentrations measured on the FRM filter with those derived from the different channels of the speciation monitor. Compare data pairs with high and low nitrate loadings, high and low temperatures during sampling, and high and low relative humidities. Examine the correlation between light absorption on FRM filters and organic, elemental, and total carbon on the speciation sampler to determine the conditions under which absorption can be used as a predictor of different carbon fractions. Compare differences with propagated measurement uncertainties and with the spatial coefficient of variation of PM _{2.5} mass and chemical concentrations derived from simultaneous measurements at different Fresno PM _{2.5} sites.
1.2. Elemental analysis of Teflon-membrane filters under a helium atmosphere does not result in a significant (>10%) loss of volatile nitrate.	Particle analysis in EPA's speciation network is considering x-ray fluorescence analysis under a helium atmosphere rather than under the conventional vacuum. The potential advantage of this modification is that volatile species, such as ammonium nitrate, may experience less evaporation than under vacuum. Disadvantages are less sensitive detection limits, added cost of helium vented between each sample batch, and degraded performance and lifetime of x-ray detectors as helium diffuses through the beryllium window. Heating of the sample by x-ray bombardment may cause losses of volatiles regardless of the surrounding atmosphere.	Compare elemental and nitrate measurements from the FRM filter analyzed by x-ray fluorescence (XRF) under vacuum with those from the speciation sample analyzed under helium, and quantify losses of potentially volatile species. Compare nitrate levels from both of these Teflon-membrane filters with the nitrate measured by a speciation monitor on a quartz-fiber filter, non-volatilized nitrate from a denuded quartz-fiber filter, and volatilized nitrate from a backup sodium-chloride-impregnated cellulose-fiber filter. Quantify any advantages to be gained from the extra expense and lower sensitivity afforded by XRF analysis under a helium atmosphere.

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Table 1.	(continued)
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Hypotheses	Background	Data Analysis and Methods and Data Used
1. Measurement Evaluation Hypotheses (continued)		
1.3. Carbon gases absorbed on quartz-fiber filters are a small (<15%) fraction of organic carbon measured on these filters.	Previous studies in central California have found organic carbon on backup quartz filters that is 10% to 50% of that on the front filters (Chow et al., 1996; Chow and Egami, 1997). This backup filter absorption was smaller when a quartz fiber denuder preceded the filter pack (Chow et al., 1993b). When this backup carbon is added or subtracted to the front filter carbon, measured mass is grossly overestimated or underestimated. The extent to which this backup carbon is a significant particle or gas artifact is currently unknown.	Compare organic carbon from the quartz-fiber backup filter with and without organic carbon denuding and quantify the maximum potential artifact that might be expected under routine speciation monitoring. Plot the ratio of backup filter carbon to front filter carbon for both channels as a function of front filter carbon and PM _{2.5} mass. Estimate potential biases to the highest and annual-average PM _{2.5} and carbon concentrations determined from common speciation monitoring. Examine variations with respect to temperature and source contributions, especially vegetative burning.
1.4. Volatilized particulate nitrate is a minor (<10%) part of particle nitrate during winter, but a major fraction of particle nitrate during other seasons.	Substantial savings in sampling and analysis costs can be attained if it can be shown that nitrate is accurately sampled with a single filter during cool winter and fall months when nitrate levels are high.	Plot nitrate and ammonium concentrations from the denuded front filter as a function of total nitrate, stratified by temperature and relative humidity during sampling. Compare nitrate from FRM and from continuous monitors with total particulate nitrate from the denuded quartz-fiber and sodium-chloride-impregnated backup filters. Specify the sampling and analysis conditions under which nitrate from non-denuded samples without backup filters can reasonably represent particulate nitrate in the atmosphere.
1.5. Volatilized particulate nitrate is a minor ($<10\%$) part of actual PM _{2.5} during all seasons.	Although larger fractions of nitrate may be lost during sampling for non-winter periods, this may be a small fraction of $PM_{2.5}$ mass under certain conditions.	Plot volatilized nitrate and ammonium concentrations as a function of $PM_{2.5}$ from different monitors, including the heated Tapered Element Oscillating Microbalance (TEOM). Determine the nature of those situations under which volatilization is more than 10% of measured mass. Add particle nitrate and ammonium to the heated TEOM mass, and compare the results with filter-based $PM_{2.5}$ to determine the extent to which TEOM volatilization is specific to ammonium nitrate.

Table 1.	(continued)
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Hypotheses	Background	Data Analysis and Methods and Data Used
1. Measurement Evaluation H	lyotheses (continued)	
1.6. PM _{2.5} mass concentrations estimated from particle size, weighted sums of chemical components, light scattering, light absorption, and light extinction, are equivalent to gravimetric mass of samples taken with a PM _{2.5} FRM sampler.	These measures have been found to be highly correlated in previous studies, but their relationships change with aerosol composition and environmental conditions. Knowing the conditions under which equivalence can be expected will expand the utility of different continuous PM _{2.5} surrogate measurements taken at other sites and lessen the need for extensive collocated measurements.	Estimate $PM_{2.5}$ mass concentrations from particle size data using reasonable assumptions about particle shape and density. Estimate $PM_{2.5}$ mass from light scattering, light absorption, or light extinction based on reasonable assumptions about particle shape, density, index of refraction, size distribution, and liquid water uptake. Estimate $PM_{2.5}$ mass based on reasonable assumptions about unmeasured hydrogen and oxygen associated with measured chemical components. Compare these mass estimates with $PM_{2.5}$ mass measured by FRMs and by the speciation monitor with volatilized components added. Explore the nature of discrepancies to determine the potential causes, in terms of particle climatology, deviations from mass estimates with differences due to collocated measurement uncertainty, climatology, and filter- based sampler differences.
1.7. PM _{2.5} and PM ₁₀ mass measurements are comparable for all measurement methods during spring and summer when the sampled aerosol is stable. Mass measurements diverge during winter and part of the spring when volatile nitrate and organics constitute a large mass fraction.	These comparisons between diverse measures of $PM_{2.5}$ and PM_{10} are needed because other sites do not have the full range of instruments at Fresno. Inexpensive, portable nephelometers will be located with portable filter samplers at satellite sites to determine spatial homogeneity. Consistent relationships between FRM PM _{2.5} and light scattering, even ones stratified by relative humidity and aerosol composition, will be needed to evaluate the zone of representation for the Fresno site.	Apply equivalence measures described in Section I to PM _{2.5} and PM ₁₀ mass measurements from FRMs, dichots, Hivol/SSI, BAM, and TEOM measurements. Stratify comparison data sets by season, temperature, relative humidity, and nitrate concentration and determine changes with aerosol composition. Calculate linear regression estimates of PM _{2.5} from light scattering and absorption measurements and examine how these differ with changing aerosol composition and environmental variables.

Table 1.	(continued)
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Hypotheses	Background	Data Analysis and Methods and Data Used
1.8. Particle number counts in integrated sub-ranges of the 0.01 to \sim 10 µm size distribution are comparable to PM _{2.5} and PM ₁₀ mass measurements with definable assumptions of constant shape and density.	This will allow different particle size measurement devices to be evaluated as potential equivalent methods for estimating $PM_{2.5}$ concentrations. The added information of particle number concentration would provide a useful long-term data base for health studies and source identification.	Examine chemical compositions from speciation measurements and compile densities for particle compositions expected in different size ranges. Assuming spherical geometry, calculate total volumes for each bin and sum to obtain mass equivalents. Apply the Section I comparison measures to determine the extent to which these estimates relate to mass measurements.
1.9. Differences between continuous PM_{10} and $PM_{2.5}$ monitors are comparable to coarse particle mass concentrations on dichotomous samplers.	Continuous measurements of coarse particle mass provide better time resolution and more frequent samples than dichotomous samplers. If biases in BAM and TEOM measurements affect the PM_{10} and $PM_{2.5}$ samples in the same way, the coarse mass fraction may be comparable to that of a dichotomous virtual impactor filter measurement.	Calculate hourly time series of differences between PM_{10} and $PM_{2.5}$ for BAM and TEOM and compare them with each other. Compare 24-hour averages of these differences with dichotomous sampler coarse mass concentrations. Examine time series plots of these differences, with propagated uncertainties, and identified periods for which agreement is good and poor. Explain disagreements in terms of aerosol composition and meteorological changes.

Hypotheses	Background	Data Analysis and Methods and Data Used
1.10 The PM _{2.5} geological component is comparable to the difference between continuous PM _{2.5} mass measurements and the sum of continuous nitrate, sulfate, and carbon concentrations (adjusted for ammonia, hydrogen, and oxygen).	Practical technology does not yet exist for the in situ quantification of soil- related elements as it does for the other major components of $PM_{2.5}$ and PM_{10} . Mass can usually be reconstructed to within $\pm 10\%$, however, with weighted sums of sulfate, nitrate, carbon, and elements when humidities are not excessive (Chow et al., 1996). If this holds true for the short-term measurements, then the geological component could be inferred from the other continuous measurements, providing complete characterization of major components at ~30 min intervals.	Calculate the difference between hourly $PM_{2.5}$ mass and weighted sums of sulfate, nitrate, and carbon for all available measurements. Propagate errors and evaluate the extent to which this difference exceeds its precision. Compare 24-hour averages of this difference with the geological fraction determined from the collocated $PM_{2.5}$ dichotomous and speciation monitors.
1.11. Bioaerosols and endotoxins constitute a constant fraction of coarse particle mass.	Aluminum, silicon, calcium, titanium, iron, and other elements have been found in reasonably uniform proportions in soils throughout the San Joaquin Valley (Houck et al., 1989). Bioaerosols such as pollen, molds, and spores and endotoxins are less likely to exhibit constant proportions to coarse particle mass and elemental concentrations.	Calculate mass ratios of elemental, bioaerosol (pollen, mold, spore) and endotoxin concentrations for coarse particle samples. Examine how these fractions vary in space and time. Identify periods for which there is substantial variation from a constant ratio and relate these to meteorological and plant-growing conditions.

Table 1.	(continued)
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Hypotheses	Background	Data Analysis and Methods and Data Used
1.12. Photoionization and wavelength-specific light absorption are correlated with organic compound concentrations.	These methods have shown good correlations with aggregates of certain organic compounds. There is good evidence that PAH on particles can be specifically photoionized (Burtscher and Schmidt-Ott, 1986; Wilson et al., 1994), although instrument response is specific to the aerosol composition. Simultaneous continuous and PUF/XAD measurements of PAH and other organic substances can be used to evaluate this relationships during the winter of 2000-2001.	Create sums of different particulate organic compound concentrations, including PAHs and compare these with photoionization methods measured over the same time periods. Apply Section I comparison measures to determine predictability or equivalence. Identify samples for which a general relationship does not apply and explain it in terms of differences in aerosol composition of environmental conditions.
1.13 Nitrogen dioxide determined by standard chemiluminescence methods is an accurate measure of actual NO ₂ concentrations for health assessments.	Winer et al. (1974) showed substantial biases in NO2 concentrations reported by compliance-based chemiluminescence monitors, especially in the eastern Los Angeles basin during summer afternoons. These biases have not been evaluated for Fresno conditions, and the presence of several detailed nitrogen measurements, including real NO2, will allow these biases to be assessed.	Apply Section I comparison measures to NOx, NOy, and NO2 concentrations.

Hypotheses	Background	Data Analysis and Methods and Data Used
2. Aerosol Characterization and Control Strategy Hypotheses		
2.1 Short duration (~5 min) spikes in particle measurements represent contributions from nearby (<500 m) emitters.	If this hypothesis is proven, it will provide new methods to determine the zone of influence of specific emitters. These zones of influence need to be known so that the spatial extent for applying emissions reductions can be determined.	Examine time series of shortest time averaged data available from continuous particle size, light scattering, light absorption, mass, and chemical specific measurements. Determine the extent to which integrating nephelometers used at CRPAQS ^c satellite sites show short-duration peaks that correspond to these variables at the CORE site. Create pollution roses (average concentration as a function of wind direction) for these averages and examine them for source directionality. Use time series analyses and frequency distributions to determine the need for more frequent sampling and for shorter duration sampling than is current practice. Calculate spatial correlations among sampling spikes of 5 min, 1-hr, 3-hr, 5-hr, 8-hr, and 24-hr measurements of light scattering using CRPAQS nephelometer measurements from satellite sites surrounding the CORE site. Calculate spatial correlations of 5-min spikes over longer-term averages to evaluate zone of influence of nearby sources.

 Table 1. (continued)

Hypotheses	Background	Data Analysis and Methods and Data Used
2. Aerosol Characterization and	nd Control Strategy Hypotheses (contin	aued)
2.2 Nearby emitters represent a small (<15%) fraction of PM _{2.5} measured at a CORE sampling site.	If a CORE site is properly sited, the spikes caused by nearby emitters should be a small proportion of the PM _{2.5} contributed by sources with urban and regional zones of influence.	From 5-min spikes over longer-term averages, estimate the incremental mass contributed by nearby sources. Plot these increments as a function of $PM_{2.5}$ and PM_{10} mass concentrations over 1-hr, 3-hr, 5-hr, 8-hr, and 24-hr periods. Describe the particle climatology for those situations under which nearby sources are a large fraction of CORE site concentrations. Determine how much $PM_{2.5}$ and PM_{10} and their chemical components change during the day and from day to day. Examine the day-to-day (24-hr average and diurnal variations of $PM_{2.5}$ and PM_{10} and their chemical components change during the day and from day to day. Examine the day-to-day (24-hr average mass and chemical concentrations. Plot PM mass, chemical components and PM precursors species. Where available, examine the 1-hr, 3-hr, 5-hr, and 8-hr average mass and chemical concentrations. Plot PM mass, chemical composition, and precursor species concentrations as a function of time for sites collecting 24-hr data. Note similarities and differences between: (1) diurnal patterns for $PM_{2.5}$ and PM_{10} and their chemical components, and assess dominant species in each size fraction by time of day for high vs. low values. Plot spatial pie charts and describe spatial patterns as a function of time of day and over a 24-hr average period (midnight to midnight). Compare episode periods to periods of lower PM concentrations as a function of time of day and location by site type or site environment. State and justify conclusions concerning: (1) differences between sites, (2) chemical composition as a function of time of day. (3) chemical composition on episode days. (4) differences between $PM_{2.5}$ and PM_{10} and precursor species as a function of the time of the day and for episode days.
2.3 The majority of ultrafine particles are from nearby (<500 m), fresh emissions sources.	Small particles with aerodynamic diameters <0.1 μ m (PM _{0.1} or "ultrafine" particles) are believed to coagulate into larger particles within a short distance of their emissions. If this is true, their numbers should increase in short duration spikes and be consistent with wind directions favoring nearby emitters, such as highly-traveled First Street.	From 5-min spikes over longer-term averages of ultrafine particles as small as 0.003 μ m measured with the Condensation Particle Counter, estimate the incremental mass contributed by nearby sources. Plot these increments as a function of PM _{2.5} and PM ₁₀ mass concentrations over 1-hr, 3-hr, 5-hr, 8-hr, and 24-hr periods. Describe the particle climatology for those situations under which nearby sources are a large fraction of CORE site concentrations. Compare ultrafine particle concentrations at the Fresno Supersite with CRPAQS non-urban background site (Angiola) to examine spatial variabilities of ultrafine particles.

Hypotheses	Background	Data Analysis and Methods and Data Used
2. Aerosol Characterization a	nd Control Strategy Hypotheses (contin	ued)
2.4 Ammonium nitrate reductions are limited by available nitric acid rather than available ammonia in urban areas during all seasons and all hours of the day.	Limited measurements of ammonium nitrate and precursors indicates that this is the case. Existing measurements do not represent short enough sample durations or all seasons of the year to determine the need for ammonia or oxide of nitrogen emission reductions.	Using continuous measurements for particle sulfate, particle nitrate, ammonia, nitric acid, temperature, and relative humidity, determine the conditions under which reducing ammonia concentrations will result in reductions of ammonium nitrate or reduce the neutralization of sulfuric acid in Fresno. Apply an aerosol equilibrium model using 1-hr average total ammonia and total nitrate concentrations (Watson et al., 1994a; Blanchard et al., 1997). State and justify conclusions about where and when ammonium nitrate concentrations are limited by ammonia levels, and when they are limited by nitrate levels, with special attention to time of day and time of year. Compare calculated ammonium mitrate concentrations with measurements and evaluate how well the equilibrium model applies in the San Joaquin Valley. Examine model sensitivities to changes in temperature and relative humidity over available sampling intervals. Determine the extent to which conclusions drawn from previous measurements for longer averaging periods and shorter sampling periods are valid under a wider variety of conditions. Plot isopleths of constant ammonium nitrate concentrations of total ammonia and nitrate. Identify the location of typical measurements on these plots and determine the ammonium nitrate concentrations would be observed. Classify each sample as ammonia or nitrate limited. For each sample, reduce each ammonium sulfate concentration by half, and to zero, examining the changes in ammonium nitrate with these reductions.

Hypotheses	Background	Data Analysis and Methods and Data Used
2. Aerosol Characterization a	nd Control Strategy Hypotheses (contin	nued)
2.5 Advanced gas and particle organic speciation measurements, coupled with elements, ions, and organic and elemental carbon fractions, consistently and accurately distinguish contributions from different types of suspended dust, secondary sulfate and nitrate, wood combustion, field burning, meat cooking, gasoline engine exhaust from cold starts, high emitters, and hot stabilized operations, diesel exhaust, and primary industrial emissions.	Watson et al. (1998b), Schauer et al. (1996), and Schauer and Cass (1998) show the possibility that this may be the case in Denver, CO; Los Angeles, CA; and in wintertime Fresno, CA. A more extensive evaluation of these approaches, combining gas and particle organic and inorganic speciation, is needed.	Using CRPAQS gas and particle organic and inorganic speciation of source and receptor samples, calculate source contribution estimates with the Chemical Mass Balance (CMB) modeling approach (e.g., Watson et al., 1997b, 1998c; Schauer et al., 1996; Schauer and Cass, 1998). Examine the temporal and spatial variation of source contribution estimates with respect to known spatial and temporal distributions of emissions and determine consistencies and inconsistencies. Plot these contributions for each sample as stacked bar charts and compare the apportionments among sampling sites and sampling periods and for episode and non-episode days. Summarize the magnitudes of source contributions at each sampling site in frequency tables. Conduct sensitivity and randomized data tests to evaluate the magnitudes of uncertainties in apportionments. Compare source contributions among nearby sites for consistencies and inconsistencies. Classify each available sample by its major contributors and determine how many cases of excessive PM concentrations are dominated by a single source type versus those that represent a superposition of sources.
2.6 Commonly measured elements, ions, and organic and elemental carbon fractions consistently and accurately distinguish contributions from suspended dust, secondary sulfate and nitrate, vegetative burning (wood and field combustion and meat cooking), gasoline engine exhaust (cold starts, high emitters, and hot stabilized), diesel exhaust, and primary industry contributions.	Watson et al. (1998b) showed that this is the case in Denver by comparing apportionments from conventional measurements that will be available from $PM_{2.5}$ speciation networks with more detailed source contribution estimates using detailed organic compounds. This needs to be generalized to another environment such as central California.	Calculate CMB source contributions using commonly measured components without the enhanced organic speciation. Compare source contribution estimates with those derived from the detailed measurements and draw conclusions about which source categories must be combined to minimize collinearity. For these categories, apply the CMB to the chemically speciated measurements taken at the Supersite and nearby sites in the urban area. Use these source contribution estimates to corroborate the zone of influence of different source types examined under previous hypotheses.

Table 1.	(continued)
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Hypotheses	Background	Data Analysis and Methods and Data Used		
2. Aerosol Characterization a	2. Aerosol Characterization and Control Strategy Hypotheses (continued)			
2.7 Gasoline engine cold starts and high emitters are the major causes of gasoline-fueled vehicle contributions to PM _{2.5} , and they cause gasoline exhaust contributions to exceed diesel exhaust contributions.	Current emissions inventories do not explicitly represent contributions from high-emitting vehicles, and inventoried diesel emissions exceed gasoline vehicle emissions. Receptor model and emissions testing evidence from wintertime Denver (Watson et al., 1998b) indicates that cold starts and poorly maintained gasoline vehicles may constitute the bulk of gasoline exhaust emissions. This finding needs to be evaluated in other environments such as central California.	Compare the proportional contributions from different source categories with similar proportions in emissions inventories, using results from the enhanced and common CMB receptor modeling. Identify discrepancies between receptor contributions and inventory estimates, taking diurnal and seasonal variations and source zones of influence into account.		
2.8. Statistical aggregates of concentration indicators for a single year deviate by less than sampling error from a three-year distribution.	Arithmetic averages, seasonal averages, maximum 24-hour concentrations, maximum 1-hour concentrations, and various percentiles may vary substantially from year to year. In particular, the CY2000 data set that will be used for Central California air quality planning needs to possess statistical indicators that are similar to previous and subsequent years to produce robust emissions reduction strategies	Calculate statistical indicators and their standard errors for each year and each observable. Apply parametric statistical tests for normal and long-normal distributions to evaluate the significance of year-to-year differences.		

Table 1.	(continued)
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Hypotheses	Background	Data Analysis and Methods and Data Used	
2. Aerosol Characterization and	2. Aerosol Characterization and Control Strategy Hypotheses (continued)		
2.9. Concentrations in continuously measured carbon fractions can be associated with different proportions of gasoline vehicle exhaust, diesel vehicle exhaust, and wood burning.	Watson et al. (1994b) found different amounts and shapes for thermograms of samples acquired from diesel and gasoline exhaust samples. Multiwavelength absorption and photoionization methods might also provide indicators of different carbon sources, especially when associated with short-duration pulses dominated by a single, nearby plume. Testing this hypothesis will make use of source characterization studies currently planned for CRPAQS and for which sponsorship will be sought in future studies.	Plot ratios of measurements from continuous carbon, PAH photoionization, light absorption, and light scattering. Determine how these ratios change when short-term spikes that might originate from nearby sources are seen. Compare ambient ratios with ratios from similar measurements in plumes from carbon emitters such a diesels, gasoline vehicles, wood stoves, and cooking.	
2.10. Annual average, seasonal average, and 98th percentile source contributions from fugitive dust, wood burning and cooking, vehicle exhaust, secondary ammonium sulfate, and secondary ammonium nitrate differ by less than $\pm 10\%$ over a three year period.	Daily chemical speciation permits source apportionment receptor modeling. Source contributions can be aggregated as statistical indicators corresponding to PM _{2.5} and PM ₁₀ concentrations that exceed NAAQS.	Calculate source contributions to 24-hour average PM _{2.5} from daily speciation monitor measurements using the Chemical Mass Balance (Watson et al., 1984, 1990, 1991, 1998c). Calculate statistical indicators for the resulting source contributions and test the statistical significance of year-to-year differences.	

Table 1.	(continued)
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Hypotheses	Background	Data Analysis and Methods and Data Used
2. Aerosol Characterization and Control Strategy Hypotheses (continue		aued)
2.11. Particle size fraction, number, surface area, and major chemical component indictors of particle concentrations are highly correlated; one indicator is equivalent to other indicators that might be specified by future air quality standards.	If all of these indicators are highly correlated, then one will be as good as another in estimating air quality. High correlations also demonstrate collinearity that make epidemiological relationships to individual variables uninterpretable.	Calculate the correlation coefficients between concentrations for data subsets stratified by time period, aerosol composition, and meteorological variables. Calculate singular values and eigenvectors for this matrix and apply collinearity measures (Henry, 1992) to determine the extent to which regression models using one or more of these variables will be biased.
2.12. Large reductions in $PM_{2.5}$ and PM_{10} mass concentrations in Central California after 1992 are due to the end of a seven-year drought rather than due to emissions reductions.	Alexis et al. (1999) show a downward trend in annual geometric average PM_{10} for the San Joaquin Valley, from a high of 70 µg/m3 in 1988 to a low of 44 µg/m3 in 1997. There is a precipitous drop between 1992 and 1993 when a seven year drought ended. Examination of the 3-year Supersite record will permit relationships to meteorology to be examined that can be applied to the long-term data record.	Examine wintertime changes in particle measurements for multi-day episodes between storms. Tabulate the frequency and duration of these episodes from meteorological recorded from 1990 through 2002. Construct weighted wintertime averages for $PM_{2.5}$ mass and chemical composition and compare these for each year of record.
3. Health and Exposure Related Hypotheses		
3.1 $PM_{2.5}$ mass concentration, surface area, and number counts are highly correlated (r2>0.8) and a measure of one is a good indicator of the other two.	It will not be possible to evaluate epidemiological effects of individual indicators if they are always highly correlated. It is more probable that there are emissions and meteorological conditions under which these indicators are correlated and other conditions under which the correlation is low.	Calculate temporal correlation coefficients stratified by particle climatology variables such as time of day, temperature, relative humidity, wind sector, ultrafine particle concentration, and PM _{2.5} concentration. Determine the conditions under which good and poor agreement will be found and the frequency of occurrence of these situations.

Table 1.	(continued)
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Hypotheses	Background	Data Analysis and Methods and Data Used
3. Health and Exposure Related Hypotheses (continued)		
3.2 Soluble transition metals are a small fraction (<15%) of total metal concentrations in PM _{2.5} .	The soluble fraction of transition metals, especially with specific valence states, is believed to be the portion that causes health. This fraction may not be accurately quantified by total elemental analysis methods that are usually applied to aerosol samples.	Plot the concentrations of soluble transition metals and total transition metals. Compare the ratios of soluble vs. total metals as a function of $PM_{2.5}$ mass. Examine the correlations among the soluble fraction of transition metals, total transition metals, and $PM_{2.5}$ mass.
3.3. Measurements at a CORE sampling site represent the minimum to which people are exposed in their neighborhoods within an urban area.	Nearby emitters, such a wood stoves and fireplaces or freeways, may add to outdoor exposure levels. These contributions have high spatial gradients that add to the urban-scale concentrations intended for quantification at CORE sites.	Determine how well the existing PM monitoring sites represent human exposure, maximum PM concentrations, and maximum source impacts by comparing measurements from nearby urban and non-urban sites with those at the Fresno CORE site. Determine spatial homogeneity and zones of representation for specific chemical components such as sulfate, nitrates, ammonium, organic and elemental carbon, and geological material (e.g., Si, Fe). Describe aerosol and precursor species sampling sites and their surroundings. Classify the spatial scale of sites (neighborhood to regional) and site types (agricultural to commercial). Evaluate the adequacy of monitoring networks for representing human exposure, maximum PM concentrations, and source influences. Use statistical analyses (such as spatial correlation analysis, cluster analysis, empirical orthogonal functions, and analysis of variance) as well as activities as a function of distance to obtain a better understanding of the relationships between/among sites and their surroundings. Plot long-term and research sites on maps with population distributions and locations of major source types/land-use types. Evaluate adequacy of site coverage and recommend new sites or site classification changes for long-term measurement sites.
3.4. Hospital and physician diagnoses of respiratory and cardiovascular ailments are as equally sensitive to changes in PM _{2.5} mass concentrations as they are to other air pollution indicators.	Current air quality standards are based on associations that can be further classified by age, race, and history of previous respiratory ailment. A variety of indicators will be available at Fresno that can be associated with different end-points.	Apply advanced statistical analysis methods to quantify relationships between health end-points and air quality indicators, taking account for inter-correlated co-factors.

Table 1. (continued)

Hypotheses	Background	Data Analysis and Methods and Data Used
3. Health and Exposure Related Hypotheses (continued)		
3.5. There is a discernible lower threshold for single and combined air quality indicators, below which no relationships with health end- points are statistically significant.	There are some indications that adverse health effects are detectable at levels lower than those specified for current air quality standards. More precise and spatially homogenous measures are needed to determine what this threshold might be for different aerosol characteristics and exposure periods.	Apply advanced statistical analysis methods to quantify relationships between health end-points and air quality indicators, taking account for inter-correlated co-factors.
3.6 Different particle chemical characteristics have different and identifiable immuno- enhancing properties that affect the symptom onset and severity of short-term reductions in lung function, asthma attacks, and cardiovascular ailments.	Asthma in children may be aggravated by elevated levels of endotoxins, bioaerosols, PAH, or diesel exhaust to a greater extent than it is by number, surface, or volume concentrations (Gielen et al., 1997).	Compare measured responses in test subjects with similar and different histories of exposure to air quality. Relate these responses to differences in air quality indicators associated with the onset and severity of distress.
3.7. Coarse particle concentrations and biologically active components show relationships to health end- points.	Since $PM_{2.5}$ is often a large fraction of PM_{10} , their levels are highly correlated. Coarse particles are not as highly correlated with $PM_{2.5}$ and they may have different and detectable harmful effects.	Apply advanced statistical analysis methods to quantify relationships between health end-points and air quality indicators, taking account for inter-correlated co-factors.
3.8. Animal (rats) exposures to different combinations of concentrated amounts of particle size, surface area, chemical, and mass characteristics result in similar indications of respiratory and cardiovascular distress.	Aerosol concentrators (Sioutas et al., 1995) are used to vary the exposure of test subjects to a variety of aerosol compositions and levels (Pinkerton et al., 1996). Reactions are often different from laboratory generated aerosols.). Compare physiological changes between rats exposed to different compositions but similar $PM_{2.5}$ mass concentrations. Identify most active agents in causing changes in cardiopulmonary response.

Table 1. (continued)

Hypotheses	Background	Data Analysis and Methods and Data Used
3. Health and Exposure Related Hypotheses (continued)		
3.9. Particles found in healthy human lungs have characteristics similar to those found in urban air over long periods of exposure.	Examination of autopsied lungs from healthy Fresno residents after accidental death shows particulate accumulations (personal communication, Dr. Kent Pinkerton, UC Davis). These particles, their location of deposit, and potential adverse affects need to be related to the actual particle characteristics that influence transfer and deposition in human airways.	Compare characteristics between autopsied human lung and ambient particles.

2. Scientific Key Findings

• Comparability between time-integrated measurements for carbonaceous aerosols (*Hypothesis 1.1, 1.3*).

Nondenuded single quartz filter packs from the FRM were compared with denuded and nondenuded quartz/quartz filter packs and nondenuded Teflon/quartz filter packs from the Andersen Reference Ambient Air Sampler (RAAS) and with nondenuded single quartz filter packs from the Desert Research Institute (DRI) Sequential Filter Sampler (SFS). Equivalence was found for PM_{2.5} mass, filter light transmission (b_{abs}), and total carbon (TC) between the Andersen single-channel FRM and RAAS speciation samplers, and for front filter TC between denuded and nondenuded channels in RAAS samplers. The average ratios of front filter carbon between the denuded and nondenuded channels in the RAAS sampler were 0.83 ± 0.19 for TC, 0.81 ± 0.20 for organic carbon (OC), and 1.01 ± 0.33 for elemental carbon (EC). The average differences for TC and OC between the undenuded and denuded channels were low (1.2 to 1.4 µg m⁻³) and are comparable to the measurement uncertainties (Watson and Chow, 2002; Chow et al., 2005a).

• Comparability between semi-continuous and time-integrated measurements for carbonaceous aerosols (*Hypothesis 1.3, 1.6, 1.7, 1.12*).

TC from the R&P 5400 continuous carbon particulate analyzer was 40–60% higher than filter carbon by thermal/optical reflectance (TOR) method, following the Interagency Monitoring of Protected Visual Environments (IMPROVE) protocol. None of the R&P 5400 versus TOR filter comparisons were comparable or predictable. These findings may be partially due to the small data set (~35 data points) used in the comparison and due to several instrument malfunctions during the experiment. A linear relationship and high correlation (r ~ 0.9) was found between b_{abs} measurements from the Teflon-membrane filter and EC measurements for EC less than 5 µg m⁻³. The absorption coefficient ranged from 11.4 ± 0.7 to 12.0 ± 0.7 m² g⁻¹ among the FRM, RAAS, and SFS samplers. PM_{2.5} b_{abs} measurements can be added to the FRM compliance monitoring network to estimate PM_{2.5} EC concentrations (Watson and Chow 2002).

The single-wavelength and seven-color aethalometers were highly correlated (r = 1) and comparable to each other, but an average of 0.11 µg m⁻³ less black carbon (BC) was measured by the seven-color aethalometer for the 880-nm channel. Diurnal BC variations showed that BC concentrations were similar between the two aethalometers between 0900 and 1600 Pacific daylight time (PDT), but disparities (>0.15 µg m⁻³ BC) occurred during late evening and early morning hours when BC concentrations were elevated. Both aethalometers met the criteria for predictability from EC on filter samplers, and they met the criteria for comparability with SFS EC measurements. Measurements of filter-based TOR EC were consistently 20–25% higher than aethalometer BC. Particle-bound PAH (relative units) were correlated with BC and EC measurements, but not sufficiently to be considered predictable.

• Organic sampling artifacts (*Hypothesis 1.3*).

OC sampling artifacts were evaluated at Fresno using the Andersen RAAS sampler with carbon-denuded and undenuded channels with Teflon-membrane and quartz-fiber filter pairs. Differences between denuded particle OC and that obtained by subtracting the quartz-behind-Teflon or quartz-behind-quartz OC from the undenuded quartz front filter were less than twice their measurement uncertainties in most cases. Particulate OC from the front filter in the denuded channel agreed most closely with the difference between undenuded front and backup quartz OC (Watson and Chow, 2002; Chow et al., 2005a).

• Equivalency in thermo/optical OC and EC measurements (*Test of measurement method for carbonaceous aerosol*).

EC results determined by simultaneous thermal/optical transmittance (TOT) corrections were 30% lower than TOR for the same temperature protocol, and 70-80% lower than TOR for a protocol with higher heating temperatures and shorter residence times. This was true for 58 urban samples from Fresno, CA, as well as for 30 samples from the non-urban IMPROVE network that are individually dominated by wildfire, vehicle exhaust, secondary organic aerosol, and calcium carbonate contributions. Visual examination of filter darkening at different temperature stages showed that substantial charring takes place within the filter, possibly due to adsorbed organic gases or diffusion of vaporized particles. The filter transmittance is more influenced by the within-filter char, whereas the filter reflectance is dominated by charring of the near-surface deposit that appears to evolve first when oxygen is added to helium in the analysis atmosphere for these samples. The amounts of charred/pyrolyzed OC (POC) and EC are also estimated from incremental absorbance. Small amounts of POC are found to dominate the incremental absorbance. EC estimated from absorbance are found to agree better with EC from the reflectance charring correction than with EC from the transmittance charring correction (Chow et al., 2004).

• The role of charring in optical correction during thermal analysis (*Test of measurement method for carbonaceous aerosol*).

A radiative transfer scheme that considers absorption, scattering, and distribution of light-absorbing elemental carbon (EC) particles collected on a quartz-fiber filter was developed to explain simultaneous filter reflectance and transmittance observations prior to and during thermal/optical analysis for carbonaceous aerosol. The model, based on extended K-M theory, was applied to study ambient filter samples from the United States and Hong Kong, China, and how they differ from each other and from reference carbon black samples. Most particles in ambient samples deposit into the top half of the filter, while resuspended carbon black particles are only found close to the filter surface. POC generated during thermal analysis reduces filter reflectance and transmittance in a fashion that suggests a uniform distribution of POC throughout the filter. When heated in oxygen, most EC evolves earlier than the within-filter-charring for certain ambient samples; this not only reflects the different natures of EC, but also prevents an unambiguous optical correction to quantify OC and EC by thermal analysis, especially when the POC/EC ratio

is large. Particle absorption in the filter is estimated for comparisons with the EC/POC measurements, suggesting a mass absorption efficiency of ~ $2.7 \pm 0.2 \text{ m}^2 \text{ (gEC)}^{-1}$ for reference carbon black and > 15 m² (gEC)⁻¹ for ambient EC, similar to previous estimates in the literature (Chen et al., 2004).

• The nitrate sampling artifacts in the San Joaquin Valley, California (*Test of hypothesis* 1.2, 1.4, 1.5, 2.4).

Evaporative loss of ammonium nitrate from quartz-fiber filters during aerosol sampling was evaluated. PM2.5 sampling was conducted using DRI SFS from December 1999 through February 2001 at three urban (Bethel Island, Fresno, and Bakersfield) and two non-urban (Sierra Nevada Foothills and Angiola) sites in the San Joaquin Valley (SJV). Non-denuded front filter non-volatilized particulate nitrate concentrations were similar throughout the SJV and followed similar temporal trends during CRPAQS. Compared with total particulate nitrate (the sum of non-volatilized nitrate from front filter and volatilized nitrate from sodium chloride-impregnated backup filter), evaporative nitrate losses ranged from less than 10% during cold months to more than 80% during warm months. In agreement with theory, evaporative loss from quartz-fiber filters in nitric acid denuded samplers is controlled by the ambient nitric acid to particulate nitrate ratio, which is determined mainly by ambient temperature. Accurate estimation of nitrate volatilization requires a detailed thermodynamic model and comprehensive chemical measurements. Volatilized ammonium nitrate was as much as 44% of actual PM_{2.5} mass (i.e., measured mass plus volatilized ammonium nitrate) in Fresno during summer. This sampling artifact has significant implications with respect to evaluating emerging standards for $PM_{2.5}$ (Chow et al., 2005b).

• Temporal variation of carbonaceous aerosol (Test of hypothesis 2.2, 2.5).

OC and EC concentrations were higher during the colder months and the trend was most pronounced at Fresno and Bakersfield, where OC and EC concentrations reached 24 \pm 3 and 8.3 \pm 1.6 µg/m³ on January 1, 2001. OC and EC displayed similar diurnal trends at the urban Fresno site during winter (December–February) and summer (June–August) and at the regional Angiola site during winter. In Fresno during the winter, OC and EC concentrations peaked at night due to residential wood combustion and the development of a nighttime inversion. During the summer, there were distinct morning and late-afternoon rush hour peaks. The winter diurnal pattern at Angiola reflected transport of secondary pollutants to this site (Watson and Chow 2002; Chow et al., 2005).

• Zones of representations of ambient monitoring site (*Test of hypothesis 2.1, 2.2, 2.5, 2.6, 3.3*).

For annual $PM_{2.5}$, sites at Bethel Island (BTI), a regional-scale transport site, and Angiola (ANG1), a non-urban site, represented an area of 15 and 20 km in radius, respectively, somewhat greater than the urban-scale Fresno Supersite (FSF) and Bakersfield (BAC) (~ 10 km). The Fresno Supersite represented very different zones of

representation for ammonium nitrate (NH₄NO₃) (20 km) and organic matter (OM, equal to OC x 1.4) (3 km), consistent with the strong gradient of OM concentration at the Suprsite. The major contrast among the three sampling sites in Fresno (FSF, Fresno residential site [FRES], Fresno motor vehicle site [FREM]) and a site in its sister city Clovis (CLO), therefore, were caused by OM rather than NH₄NO₃. Site density down to the neighborhood-scale is necessary to resolve spatial variability of OM in this urban area. Despite being located downtown, the BAC site appeared to be representative of the entire city in terms of annual PM_{2.5}, NH₄NO₃, and OM. OM accounted for a minor fraction of PM_{2.5} at ANG1, and therefore the zone of influences for PM_{2.5} and NH₄NO₃ are similar in magnitude. The BTI site is situated in a regional-scale transport environment, so it is influenced by nearby cities such as Stockton and Sacramento. It represented a consistent zone (> 10 km) for PM_{2.5}, NH₄NO₃, and OM (Chow et al., 2005c).

• The influence of transport and mixing on the diurnal variation of PM_{2.5} species (*Test of hypothesis 2.4, 2.5, 2.6, 2.11*).

A winter $PM_{2.5}$ episode that achieved a maximum 24-h average of 138 $\mu g\ m^{\text{-3}}$ at the Fresno Supersite in California's SJV between January 2 and 12, 2000 is examined using 5-min to 1-h continuous measurements of mass, nitrate, BC, particle-bound PAH, and meteorological measurements. Simultaneous measurements at satellite sites showed that the Fresno Supersite represented PM_{25} within the city, and that half or more of the urban concentrations were present at distant, non-urban locations unaffected by local sources. Most of the primary particles accumulated during early morning and night, decreasing when surface temperatures increased and the shallow radiation inversion coupled to a valleywide layer. When this coupling occurred, nitrate levels increased rapidly over a 10-30 min period as BC and gaseous concentrations dropped. This is consistent with a conceptual model in which secondary aerosol formation above the surface layer and is effectively decoupled from the surface for all but the late morning and early afternoon period. Primary pollutants, such as OC and EC, accumulate within the shallow surface layer in urban areas where wood burning and vehicle exhaust emissions are high. Such a model would explain why earlier studies found nitrate concentrations to be more homogeneously distributed among widely separated sites in urban areas, as winds aloft of 1 to 6 ms⁻¹ could easily disperse the elevated aerosol throughout the valley (Watson and Chow, 2002).

• Particle size relationships at the Fresno Supersite (*Test of hypothesis 1.8, 2.1, 2.3*).

Measured particle sizes and concentrations varied diurnally with smaller particles associated with morning rush hour emissions. Higher large particle and BC concentrations and larger mean diameters during weekday evenings may be related to a combination of emissions from residential wood combustion and motor vehicles. The observed decrease in the ratio of ultrafine (UF) to number particle concentration after the morning rush hour is consistent with losses expected from particle coagulation. The Particle light scattering (b_{sp}) estimated using Mie theory from the measured size distributions was systematically lower than measured dry b_{sp} during winter, although the

precision was very high ($r^2 = 0.93$). During the following summer, however, measured and estimated b_{sp} agreed much more closely. This is attributed to a reduction of particle size caused by evaporation of NH₄NO₃ and possibly organic material in the heated shelter during winter. Nonetheless, the results demonstrate systematic and consistent relationships among particle concentrations and other chemical and physical parameters at the Fresno Supersite. Thus, long-term continuous particle size measurements are expected to be useful in evaluating air-quality and public health issues (Watson et al., 2002).

• Variations of nanoparticle concentrations at the Fresno Supersite (*Test of hypothesis 1.8, 2.3, 2.11*).

An Nano Scanning Mobility Particle Sizer (NSMPS) was deployed at the Fresno Supersite in August 2002. The NSMPS, a standard SMPS, and a Lasair optical particle counter (OPC) were used to measure the particle size distribution from 3 nm to 2 μ m from August 2002 through July 2003. These data were examined with a focus on variations of nanoparticle size and number concentration. Nanoparticle concentrations and the ratio of nanoparticle to total particle concentration were inversely related to particle surface area from 50 to 1000 μ m² cm⁻³. The highest nanoparticle concentrations were seen during the warmer months, i.e., spring, summer, and early fall. Nanoparticles were also present in high concentrations, up to 14,390 on an hourly basis, with increasing motor vehicle emissions during the morning rush hours.

In contrast to Atlanta (Woo et al., 2000), where fresh photochemical production resulted in nanoparticle concentrations exceeding 10^5 cm⁻³, 5 min average nanoparticle concentrations associated with peaks in solar irradiance never exceeded 24,415 cm⁻³ in Fresno. Further, an increase in number concentration with decreasing size below 10 nm was almost never observed in Fresno. This phenomenon constituted evidence for new particle production in Atlanta. This suggests that photochemical particle production may have occurred at a higher altitude followed by mixing to the surface or that the fresh particle production rate was smaller, with respect to the loss rate by coagulation with existing particles, than it was in Atlanta. This is consistent with much lower sulfur concentrations and relative humidity (RH) in Fresno than Atlanta (Watson et al., 2005).

• Levoglucosan in PM_{2.5} at the Fresno Supersite (*Test of hypothesis 2.10*).

Analyses of $PM_{2.5}$ samples collected at the Fresno supersite have resulted in the discovery of high levoglucosan concentrations. Concentrations ranged from less than 0.01 μ g/m³ during the spring and summer months to 4.05 μ g/m³ during the winter. This indicates an increase of biomass burning during the winter contributing to ambient air mass measurements in excess of the PM_{2.5} and PM₁₀ standards. Because of the lack of quantitative source profiles in the Fresno area, the actual contribution of burning to PM_{2.5} mass concentrations can only be estimated (Poore, 2002; Reinhart et al., 2005).

3. Knowledge Gaps

• The organic sampling artifact remains not definitive.

Two different types of organic diffusion denuders were used, but no difference in denuded quartz backup filter OC was found between the XAD- and Charcoal impregnated filters (CIF)-denuded samples. The average positive OC artifact was $1.62 \pm 0.5 \ \mu g \ m^{-3}$ for the nondenuded Teflon/quartz channel and $1.12 \pm 0.91 \ \mu g \ m^{-3}$ for the nondenuded quartz/quartz channel. With preceding denuders, the average negative artifact was $0.61 \pm 0.5 \ \mu g \ m^{-3}$. If the denuder removes most of the adsorbable organic gases, then ~ 9% of OC from the front filter is measured on the backup filter and is due to volatilization of carbon particles from the front filter. Without a denuder, ~17% of the OC on the front filter is measured on the backup filter, which means the quartz filter adsorption artifact exceeds the particle volatilization artifact by ~ 50%. These results are not definitive, however, owing to differences in gas-particle equilibrium with different sampling configurations and different environments. In addition to the 15–20% random errors, Huebert and Charlson (2000) estimated that systematic errors are 30–50% for positive sampling artifacts and 50% for negative sampling artifacts. More research is needed to better evaluate positive and negative carbon artifacts.

• Need development of continuous carbon analyzer.

Collocated comparisons of a year's worth of data from the Fresno Supersite provide confidence that: 1) adequate collocated precisions and comparability can be achieved among filter mass and carbon measurements if adequate quality assurance/quality control is applied, and 2) continuous measurements such as aethalometer BC can be used in conjunction with less frequently sampled filter samplers. Improvements in the in-situ continuous thermal evolution carbon analysis methods are needed in order to obtain measurements that are comparable to filter-based carbon measurements.

• Need more detailed modeling of spectral absorbability of EC in the air and within the filter.

Whether the within-filter-charring is due to adsorbed organic gases, diffusion of vaporized particles during analysis, or liquid organic particles warrants further investigation. Correcting for charred OC using laser reflectance or transmittance is uncertain due to unknown absorption efficiencies and different spatial distributions of POC and EC within quartz-fiber filters. The reflectance correction could be less biased by POC within the filter than is the transmittance correction. The simultaneous monitoring of reflectance and transmittance, when coupled with the extended K-M radiative transfer theory, provides additional information on the charring/combustion process within a quartz-fiber filter. This extended K-M theory will also be useful when considering multiple-wavelength reflectance and transmittance measurements that may better differentiate EC and POC.

• Need to measure more parameters in the current network.

Every day $PM_{2.5}$ sampling showed that many episodes are missed by commonly applied sixth-day monitoring, even though quarterly averages and numbers of US air quality standard exceedances are adequately estimated. Although Fresno Supersite measurements are consistent with the current conceptual model, they do not prove it. A more extensive upper air meteorological measurement network, continuous BC and nitrate at other urban and rural areas, as well as above and below the shallow nighttime and morning surface layer, are needed to confirm it and to determine the extent to which emissions mix throughout the SJV.

4. Technical and Economic Feasibility

The Fresno Supersite provides: 1) a long-term record of simultaneous advanced particle measurements that includes a large range of concentration levels, particle sizes, and aerosol compositions, suitable for temporal, spatial, and trend analyses; 2) supportable conclusions about specific hypotheses concerning measurement method performance, causes of excessive pollution levels, and effects on health; 3) linkages and collaboration among air quality scientists, toxicologists, epidemiologists, exposure specialists, and clinicians that better integrate and communicate their scientific findings; 4) a research infrastructure in Fresno at the First St. site to support other studies during and after Supersite monitoring; and 5) publications and reports that support local, state, and national decision-making related to standard setting and pollution controls.

Carbonaceous aerosol still represents the largest uncertainties in the ambient particulate measurements. This study examines several different carbon measurement methods that were collocated at the Fresno Supersite to determine their comparability for TC, OC, and EC. The potential sampling artifacts and analytical uncertainties are investigated in detail. This comparability is important because several of these methods are used individually at diverse locations throughout California's San Joaquin Valley and the nation to better understand the causes of excessive $PM_{2.5}$.

The problem of air quality degradation at Fresno and San Joaquin Valley where the Supersite is located is unique in many aspects, compared with other $PM_{10}/PM_{2.5}$ non-attainment areas in the country. Ammonium nitrate represents a larger fraction of PM mass than the rest of the country, due to low-sulfur fuels for mobile and stationary sources. Highly elevated organic aerosol concentration was observed around urban areas in winter. Many observations in the Supersite study provide insights into the source of particles, the scheme for secondary aerosol formation, and the transport and mixing of particles throughout the valley. Subsets of Fresno Supersite measurements are comparable to similar measurements from other Supersites established nationwide and to other measurements from additional sites and special studies in Central California. Specifically, the non-urban Angiola Tower site had nearly identical measurements that allow differentiation of regional from urban contributions.

Fresno Supersite measurements aim to establish relationships between complex and less comprehensive measurements such as those acquired from speciation monitors, FRMs, and battery-powered MiniVol monitors. These data will be available over a wider spatial scale, both in Central California and throughout the United States. A better understanding of where and when these more widely available measurements are adequate surrogates for more complex measurements will provide opportunities for comparisons and contrasts.

Instruments which offer continuous monitoring offer the potential of reduced labor costs and produce data with high time resolution (e.g., data recorded every 5 minutes). They can provide near real-time data, which is important for the forecasting and reporting of hazardous air quality episodes. Frequent and accurate data assists regulatory agencies with their forecasting and provides information needed to select appropriate control measures (e.g., wood burning restrictions). Forecasting can also influence the public's behavior which can have positive health and economic impacts. For example, people with respiratory illness are advised to limit their outdoor activity during high PM episodes. These advisories can reduce the number of people hospitalized due to respiratory distress which impacts the economic health of a community.

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6. Appendices

- A. Electronic versions of papers in PDF format for book chapters and journal articles; Power Point or PDF for Presentations
- B. Quality Assurance Final Report (See Atlanta Supersites Project, as example; <u>http://www.epa.gov/ttn/amtic/qualitypp.html</u>, dated 3-27-02)
- C. Receptor Models

Fresno Supersite Final Report

Appendix A: Comprehensive Bibliography

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March 31, 2005

APPENDIX A

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Fresno Supersite Final Report

Appendix B: Quality Assurance

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March 31, 2005

APPENDIX B

QUALITY ASSURANCE

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I. OVERVIEW OF QUALITY ASSURANCE

This section describes how the accuracy, precision, and validity of the Fresno Supersite measurements will be determined through a combination of calibration, systems audit, performance audit, and on-site performance test activities. It also refers to other parts of the technical narrative that describe hypotheses to be tested and the testing methods. Observables measured, the equipment and methods applied, sample frequency and duration, and measurement period are summarized in Table 7-1.

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 (QA) integrates quality control and quality auditing to determine these four attributes of each environmental measurement. QC prevents, identifies, corrects, and defines the consequences of deviations that might affect the precision and accuracy, and/or validity of the measurements. QA makes certain that QC activities are being carried out, evaluates accuracy against independent and traceable standards, acquires the information generated by QA activities, defines how the measurement attributes are determined and reported, and creates data qualification statements for data analysts.

Phase 1 QA is being provided by the ARB's Quality Assurance Branch and by a separately-sponsored (~500K) CRPAQS QA group. The last CRPAQS audit will occur during January, 2001. Primary responsibilities for Phase 2 Fresno Supersite QA will be carried out by the College of Engineering Center for Environmental Research and Technology (CE-CERT) at the University of California at Riverside (UCR). The ARB's Quality Assurance Branch will continue to provide independent QA for the flow rate, gas, and meteorological measurements.

QA guidance has been provided by the North American Research Strategy for Tropospheric Ozone (Hook et al., 1998), consistent with guidance from U.S. EPA (1998a). A Quality Integrated Work Plan (QIWP) will describe measurements, QC, and QA activities. This QIWP will be supported by detailed Standard Operating Procedures (SOPs) describing each measurement process (U.S. EPA, 1995). SOPs include: 1) summary of measurement method, principles, expected accuracy and precision, and the assumptions for validity; 2) materials, equipment, reagents, and suppliers; 3) individuals responsible for performing each part of the procedure; 4) traceability path, primary standards or reference materials, tolerances for transfer standards, and schedule for transfer standard verification; 5) start-up, routine, and shut-down operating procedures and an abbreviated checklist; 6) data forms; 7) routine maintenance schedules, maintenance procedures, and troubleshooting tips; 8) internal calibration and performance testing procedures and schedules; 9) external performance auditing schedules; and 10) references to relevant literature and related SOPs.

II. ACTIVITIES TO BE PERFORMED/HYPOTHESES TO BE TESTED

Hypotheses to be tested are stated in Section D.1. Measurement activities are described in Section D.2 with data analysis activities. Expected results and benefits are detailed in Section D.4. Simultaneous health studies are specified in section D.5.

QA activities include: 1) assembling, reviewing, and archiving SOPs; 2) summarizing QC and QA procedures with measurement descriptions in a QIWP; 3) specifying primary, calibration, performance test, and audit standards; 4) specifying data reporting conventions; 5) conducting systems audits of field, laboratory, and data management systems; 6) conducting performance audits of field and laboratories; and 7) preparing data qualification statements.

Several of the methods described in Table 7-1, especially for gases and meteorological measurements, are well-established with commonly available transfer standards for calibration and auditing. Many of the *in situ* measurement systems are being used for the first time in a long-term program and their reliability is not proven under these conditions. It is desired to have >95% data recovery with precisions $<\pm10\%$ for values exceeding ten times lower quantifiable limits. The goal for accuracy, as determined by comparison with independent traceable standards, is to be within a single standard deviation of precision for each measurement. These goals have been achieved in previous studies for *in situ* gas monitoring, filter sampling/laboratory analysis of elements and ions, and for meteorological observables. Particulate carbon and organic sampling and analysis methods have not shown as good accuracy and precision; standards and methods to establish these measurement attributes need to be developed as part of this project. Although continuous particle size measurements have been taken in special studies, practical methods to evaluate their performance in long-term studies are still lacking, as are quantitative estimates of their accuracy and precision.

The QA Manager will establish methods to quantify accuracy, precision and validity for each piece of data entered into the project data base. These attributes will be evaluated to produce a data qualification statement related to each data analysis activity. The statement will include data completeness, values above lower quantifiable limits, relative precision as a function of concentration, accuracy as determined by performance audits, and frequencies of data validation flags. It will also review current literature, and comparisons made in this study, to evaluate the response of different measurements systems to changes in environmental variables and aerosol composition. The data qualification statement will define the level of signal in an environmental cause that is needed to exceed the noise of the measurements system; it will be used by data analysts to evaluate the extent to which sought relationships are real or are an artifact of the measurement process.

III. STUDY DESIGN

Section D.3 described previous work performed in the Fresno area that was examined in selecting the monitoring locations and times. Ongoing and planned health studies were described in Section D.5 that complement Supersite measurements. The Fresno airshed is not affected by heavy industry. It contains a large, self-contained population center surrounded by low population farming activities. Particle pollution levels are fairly homogeneous throughout the metropolitan area. Fresno provides an opportunity to evaluate the nature and effects of

elevated ammonium nitrate, oxidized metal states, various carbon components, coarse particles, and associated toxic substances.

IV. SAMPLE HANDLING AND CUSTODY PROCEDURES

Sample handling, chain-of-custody, and archiving are specifically treated in SOPs. Most of the measurements specified in Table 7-1 are *in situ*, with instruments located in environmentally controlled shelters. To minimize sampling losses or changes and to promote comparability among these instruments: 1) sample inlet lines are as short as possible by locating instruments close to the shelter ceiling; 2) inlets are at a common height above rooftop level (~1.5 m), equivalent to the height of FRM inlets; 3) sample lines are of conducting material with straight or gently curving entries to instruments to minimize particle losses; and 4) sample line diameters are as small as possible to minimize residence time that might causes changes in temperature and humidity.

Filter, denuder, and absorber sections from particle monitors are prepared in clean laboratories and shipped to and from the field by overnight transport in cooled (<4°C) containers containing temperature indicators. Samples are stored in on-site refrigerators before and after sampling. Shipments are coordinated between the field and laboratory. Sample identifiers are bar-coded to indicate sample type, analysis type, and sampling time and location. These identifiers are entered into field and laboratory data acquisition systems to track sample status at any time during the project. The QA Manager will review chain-of-custody processes and data throughout the project and recommend improvements.

V. SAMPLE ANALYSIS METHODS

Table 7-1 identifies the sampling and analysis methods and Section D.2 describes how these methods are applied to Supersite measurements. Several common quality control activities take place for all analyses: 1) acceptance testing for contamination of substrates, reagents, extraction vials prior to use; 2) field and laboratory blank designation and analysis to determine blank levels and variability; 3) periodic performance tests of zero and span values for field and laboratory instruments to determine reproducibility and calibration drift; 4) periodic multi-point calibrations in the range of ambient concentrations to determine linearity and concentration relationships; and 5) data validation flags for field and laboratory operations that indicate deviations from procedures. Results from these common quality control activities are compiled into a separate data base and used to develop the data qualifications statement.

VI. CALIBRATION AND PERFORMANCE EVALUATION PROCEDURES

Several types of standards are needed for calibration, auditing, and performance tests. Primary standards are well characterized and protected, with stable concentrations to which all other standards are traceable. Transfer standards are often more easily produced or commonly available and are traceable to primary standards; these are used for calibration, performance testing, and auditing. The same standards can be used for calibration and performance testing, but audit standards should be independently traceable to primary standards. Performance tests may measure instrument electronic response rather than response to a specific value of an observable when transfer standards for the primary observable are lacking. Table 7-3 identifies primary and transfer standards and the frequency of application for calibration, performance testing, and auditing. Methods for presenting these standards to instruments depends on the instrument audited. Flow rates are relatively simple to verify, but evaluating continuous monitor response to particle size is impractical under field conditions.

The CE-CERT aerosol generation and measurement systems coupled with its environmental chamber will be used as the primary standard for particle size measurements. Laboratory calibration determines instrument response to particles of known size, composition, and concentration. Differential Mobility Analyzers (DMA) (Liu and Pui , 1974) generate monodisperse calibration aerosols in the 0.01 to 1 μ m diameter range with removal of multiply charged particles in the upper end of this range (Romay-Novas and Pui, 1988; Gupta and McMurry, 1989). The size of the classified particles depends on flow rates, classifying voltage, and geometrical factors (Knutson and Whitby, 1975), as confirmed by electron microscopic measurements at the National Institute of Standards and Technology (NIST) as part of a 0.1 μ m particle Standard Reference Method (Kinney et al., 1991). Particles produced with a DMA typically vary by ±10% about a mean size that is accurate to within ±5%.

A Vibrating Orifice Aerosol Generator (VOAG) (Berglund and Liu, 1973) generates monodisperse spherical droplets in the 1 to 10 μ m diameter range from an oleic acid in ethanol solution. Particle size is determined by flow rate of the liquid through the vibrating orifice, vibration frequency, and the concentration of nonvolatile solute. Microscopic analysis (Berglund and Liu, 1973) shows that particles by the VOAG are uniform to within ±1.4% and are routinely within ±2% of the expected size.

Particles generated by these instruments are presented to the field instruments and simultaneously monitored by a laboratory Condensation Particle Counter (CPC) and an aerosol electrometer. CPC counting efficiencies are close to 100% (Stolzenburg and McMurry, 1991; Zhang and Liu, 1991) for the size ranges and concentrations expected at the Fresno Supersite. At least 1,000 particles for each measurement are counted so that statistical counting errors are <3% (square root of count number). Aerosol electrometer uncertainties (Liu and Pui , 1974) depend on signal-to-noise, which is determined by aerosol concentration. Laboratory concentrations are generated with signal-to-noise levels of ~100, resulting in calibration biases of <2%. Pressures that affect particle classification by the DMA are monitored to within $\pm 0.5\%$ of full scale. For atmospheric pressures encountered in Fresno, the maximum error is $\pm 5\%$.

Calibration, performance testing, and auditing methods for laboratory operations are largely based on the preparation of standard solutions from mineral salts. NIST does not provide these types of standards. Standard solutions in a large range of concentrations are available commercially for inorganic monoatomic and polyatomic ions.

Gas and meteorological monitors are often used in compliance networks, as common procedures and standards have been developed for their calibration and auditing. Independent ARB staff perform these audits each year, along with audits of other air quality and meteorological monitors in the state. Some of the novel measurements in Table 7-1 will be evaluated by comparison with other measurements that have traceable standards and audit trails. The QA Manager will conduct a field and laboratory systems audit, a laboratory performance audit and/or interlaboratory comparison, and three field performance audits. Systems audits examine all phases of measurement and data processing to determine that the SOPs are followed and that operational staff is properly trained. The systems audit is intended to be a cooperative assessment resulting in improved data, rather than a judgmental activity. Performance audits establish the extent to which data specifications are being achieved in practice and evaluate measurement accuracy against independent standards. The field systems audit will be conducted near the beginning of the project after all equipment is installed and operating. It will be followed by the first field performance audit. These audits will identify deficiencies and implement remedial actions. Subsequent field performance audit results will be used to define accuracy of field measurements.

Laboratory audits will consist of presentation of standards with known concentrations to each laboratory process. These will be analyzed according to normal procedures and the results will be compared with the standard values. As shown in Table 7-3, reliable transfer standards can be obtained for mass, elements, ions, and total carbon. Common standards are not available for organic and elemental carbon.

Inter-laboratory comparisons will operate on the exchange of portions of the same filters or sample extract. Mass, elemental, ion, and carbon analysis can all be performed on portions of the same filter. DRI routinely conducts interlaboratory comparisons with laboratories at the ARB and the South Coast Air Quality Management District.

VII. DATA REDUCTION AND REPORTING PROCEDURES

The data management system will consist of a set if inter-related files with referential integrity in a Microsoft SQL Server maintained by the ARB. The data base will be available over the internet and will be integrated with other particulate, air quality, visibility, and meteorological data bases throughout Central California. Data from the Fresno Supersite and satellite sites will be submitted to this data base. A separate data base will be compiled for the Supersite measurements that will be made available via a DRI web server and through EPA and NARSTO centralized data bases. The following types of tables will be included in the project database:

- **Measurement locations:** Each measurement location is identified with a unique alphanumeric site ID accompanied by its name and address, coordinates, elevation, its primary operator, and a summary of measurements taken at the site for different monitoring periods. Coordinates are determined with GPS using map basis NAD-83 (Federal Aviation Administration convention). The GPS time stamp is recorded to correct coordinate deviations. A long-term GPS monitor at the Fresno Supersite will allow other investigators to adjust their GPS readings for drifts induced by the U.S. Department of Defense for security purposes.
- Variable definitions: Each variable is assigned a unique code that is accompanied by its definition, units, averaging time, measurement method, applicable temperature and pressure adjustments, and data reporting format.

- **Data validation flags:** Flags specific to each measurement are translated into a common set of validation flags that are carried with each data point. These are currently being defined by EPA for its speciation program, and this will be a starting point for Fresno Supersite data validation flags.
- **Data tables:** Basic data tables are constructed in normalized formats that have the same structure for different types of data. Each record contains the site code, sample date (MM/DD/YYYY), sample time (HH:MM:SS PST), variable code, measurement value, measurement precision, validity code, and validation level. These files will be transparent to most users and can be easily manipulated into convenient data analysis forms. Missing or invalid measurements contain a "NULL" value. Modern data management software permits this in place of the –99 (and variants) previously used. Separate tables are produced for different averaging times and for non-uniform data sets.
- Validation tables: Detailed information on specific samples indicating the nature of the data qualification. These tables also contain the validation level assigned to each data item.

Data validity levels are designated in the validation tables for different stages of data acquisition and interpretation. Level 0 designates data sets downloaded from a field instrument that have not been examined. These measurements are used to evaluate instrument performance and to forecast conditions for special experiments. Level 0 data are not used for interpretive purposes. Level 1 data has been evaluated by the measurement investigator prior to submission to the data base. Values are removed for instrument downtime and performance tests, adjustments for calibration deviations are applied, extreme values are investigated, internal comparisons are made, blanks are subtracted, precisions are estimated and propagated, and appropriate data qualification flags are assigned. These are submitted to the Data Manager who applies several additional tests. For sequential measurements, jump tests, standard deviation tests, and extreme value tests often identify values that need to be investigated. Level 2 data have completed intercomparison tests between data sets. These tests often result in the investigation of several samples that do not follow the same pattern as other measurements. These samples are investigated, sometimes re-analyzed, and re-designated as valid, invalid, or suspect as a result of the investigation.

Level 3 validation occurs after measurements are used to test hypotheses and values that are found contradictory to other values have been investigated. The quality of these measurements is especially important as they often indicate large deviations from conventional wisdom that should not be confused with measurement error. 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 that would normally track the values of other variables in a time series; and 3) values for observables that would normally follow a qualitatively predictable spatial or temporal pattern.

The QA Manager audits the integrity of the data base by randomly selecting data sets and tracing them through the data management system to their final values in the finished data base.

Unit conversion, sample times, site and variable codes, and data validation flags are applied manually, and the results are compared with data extracted from the internet-based data set.

VIII. INTENDED USES OF THE DATA

Data analysis activities are described in Section D.2. For QA purposes, substantial comparisons among measurements will be made to determine their predictability, comparability, and equivalence. Although the different observables measured are quite diverse, it is possible that they may be highly correlated owing to their quantification of related particle properties or to large fluctuations caused by emissions and meteorology. Relationships between variables will depend on the composition of the aerosol as well as meteorological conditions. Measures of predictability, comparability, and equivalence are applied to data sets stratified by aerosol composition and season. Predictability requires a consistent and reliable relationship between measurements, even if they are of different quantities. Light scattering or light absorption measurements are examples of continuously measured particle properties from which PM_{2.5} concentrations might be predicted. Comparability can be established between monitors that ostensibly measure the same observable, but with different principles. PM_{2.5} mass acquired from the BAM, TEOM, FRM, DICHOT, and speciation monitors are expected to be comparable, and if they are shown to be so, they can be used interchangeably in data analysis. Equivalence is a regulatory term that allows a method to be designated as Federal Equivalent Method (FEM) applicable to compliance monitoring. Equivalence is more demanding than predictability or comparability in that it requires demonstration of comparability within high tolerances over a wide range of concentration loadings and measurement environments.

Several empirical and statistical measures are applied to evaluate predictability and equivalence (Mathai et al., 1990). Linear regression is most commonly used and is the requirement for FEM relationships with FRMs. Regression slopes and intercepts with effective variance weighting (Watson et al., 1984) for each set of paired measurements are evaluated with their standard errors. The effective variance weighting includes the precisions of both variables in the calculation and bases the standard errors on them. When the slope equals unity within three standard errors, when the intercept does not significantly differ from zero within three standard errors, and when the correlation coefficient also exceeds 0.9, the measurements are considered comparable. When the correlation coefficient exceeds 0.9 but the slope and intercept criteria are not met, the dependent variable is predictable from the independent variable.

Other comparison measures include average ratios and standard deviations, ratios of averages, and the distribution of differences (X minus Y) for $<1\sigma$, 1σ to 2σ , 2σ to 3σ , and $>3\sigma$ precision intervals. These measures indicate the extent to which long-term averages are more or less equivalent than individual values and whether or not the majority of differences are within stated uncertainty intervals.

IX. PROCEDURES TO EVALUATE SUCCESS OF PROJECT

Success of the project will be evaluated in terms of: 1) accuracy, precision, validity, and completeness of acquired data; 2) extent to which data can be used to test stated hypotheses; 3) confidence of conclusions regarding hypotheses; 4) consistency of Fresno Supersite measurements with those from other California sites and other Supersites; 5) integration with

other monitoring networks and research studies; 6) leveraging of Supersite resources with those from other agencies; and 7) relevance of study conclusions to Supersite program objectives.

A final report by the Principal Investigator will discuss accomplishments with respect to each of these areas. The first topic will be assessed by the data qualification statement described above. The second topic will be assessed by the data analysts as they use the acquired measurements to test the hypotheses. Part of the success in using the data will be the ratio of data manipulation vs. data analysis time. The web-based data delivery system must allow all analysts to quickly integrate measurements that are most convenient for their tasks. Data quality information must be quickly available for consultation when inconsistencies with conceptual models are found.

Confidence in study conclusions will be evaluated according to the following criteria: 1) high confidence: a) low uncertainty in the data or data analysis approach, or b) more than one independent analysis approach, each of which has moderate uncertainties; 2) medium confidence: moderate uncertainty in the data or data analysis approach and independent analysis approaches were not applied; and 3) low confidence: large uncertainty in the data or data analysis approach and independent analysis approaches were not applied; and 3) low confidence: large uncertainty in the data or data analysis approach and independent analysis approaches were not applied or were contradictory. These ratings were applied by each investigator and modified under scrutiny by all investigators in the Northern Front Range Air Quality Study (Watson et al., 1998b). Decision-makers found it useful for scientists to express their own levels of belief in the outcome of their study.

Consistency of Fresno measurements with those from other Supersites will be included in the data qualification statement. This will include a table of overlapping observables and measurement periods acquired at the different sites that will facilitate generalization of Fresno conclusions to those of other locations. Substantial integration with other monitoring networks and research studies, as well as leveraging opportunities, have been identified in Section D. The extent to which these opportunities are realized will be evident in the project reports and publications. These reports and publications will be judged by the project sponsors concerning their relevance to Supersite objectives.

X. EXTERNAL AND PEER REVIEW

An advisory panel for the Fresno Supersite operations will be formed that includes the following members: 1) principal investigators for concurrent health studies; 2) officials from state and local air quality planning agencies; 3) EPA headquarters representatives; and 4) other project sponsors. This group will review the QIWP and other project plans and provide recommendations on how data quality might be enhanced, the extent to which Supersite measurements can be used for planning purposes and health studies, and evaluation of study findings. Scientific papers will be submitted to external peer review, and the resulting comments will be addressed in finally published papers. The Principal Investigator and selected task leaders will report on progress and results at periodic meetings of Supersite investigators. These presentations will be structured to obtain feedback and experience from similar projects taking place in other U.S. cities.

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Appendix C: Receptor Modeling

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March 31, 2005

Receptor modeling information is being processed as a part of the California Regional $PM_{10}/PM_{2.5}$ Air Quality Study (CRPAQS) and will be released with the CRPAQS final report.