IITRI
IIT Research Institute
10 West 35 Street, Chicago, Illinois 60616
312/567-4000

5 October 1989

Simpson Weather Associates, Inc.
809 East Jefferson St.
Charlottesville, VA 22902

Attention: Mr. G.D. Emmitt

Subject: **Microscopical and Chemical Analysis of Six PM10 Samples**
Letter Report on IITRI Project No. C08025

Dear Mr. Emmitt:

Six samples of airborne particulate less than 10 um in aerodynamic diameter (PM10) were submitted by Simpson Weather Associates for analysis. The samples were collected near the Dominion Terminal Associates' coal storage facility in Newport News, VA. Four of the samples were submitted for full PM10 analysis (microscopical, chemical and low temperature ashing analysis). The primary goal of the full analyses was to determine the concentration of coal particles in the PM10. Two samples were submitted only for chemical analysis to determine the contributions made by sulfate and nitrate salts to the PM10. None of the samples represented an exceedance of the USEPA primary air quality standard for PM10 of 150 ug/m3.

1. SUMMARY OF RESULTS

Full Analysis Samples—

Raw coal represented 49% (40 ug/m3), 44% (52 ug/m3) and 27% (16 ug/m3) of the sample mass in the three full analysis samples with the highest PM10 levels, and 19% (4 ug/m3) of the sample mass in the low PM10 concentration sample (see Table 1). The mean particle size of the coal in each of the four samples was 8 um.

Ammonium sulfate comprised 8% (7 ug/m3), 27% (32 ug/m3) and 37% (21 ug/m3) of the PM10 in the higher level samples, and 15% (3 ug/m3) of the PM10 in the low level sample. Ammonium nitrate occurred in relatively low concentrations, representing up to 1% (<1-1 ug/m3) of the PM10 in the higher level samples and 4% (1 ug/m3) of the PM10 in the low level sample.

Minerals were a significant component of only one of the four samples, contributing 35% (28 ug/m3) of the PM10 in one of the higher level samples, and up to 1% (<1-1 ug/m3) of the aerosol in the other three samples. Soil-derived mineral types, principally mica, clay and quartz, were predominant.

Particles identified as "other carbon" in the samples consisted of vehicle exhaust from gasoline and diesel engines, oil soot and rubber tire fragments.

Table 1. SUMMARY OF MICROSCOPICAL ANALYSIS RESULTS

Component	Filter #12		Filter #55		Filter #62		Filter #45	
	%	ug/m3	%	ug/m3	%	ug/m3	%	ug/m3
Coal	49	40	44	52	19	4	27	16
Ammonium sulfate	8	7	27	32	15	3	37	21
Ammonium nitrate	1	1	<1	<1	4	1	<1	<1
Minerals	35	28	1	1	<1	<1	<1	<1
Other Carbon	3	2	14	17	52	10	16	9
Biologicals, paper	3	2	13	15	10	2	19	11
Glassy flyash	<1	<1	<1	<1	<1	<1	<1	<1
PM10	—	80.6	—	116.7	—	20.1	—	58.4

These components together represented 3%, (2 ug/m3), 14% (17 ug/m3) and 16% (9 ug/m3) of the aerosol in the higher concentration PM10 samples, and 52% (10 ug/m3) of the aerosol in the low concentration PM10 sample. The majority of the "other carbon" was comprised of vehicle exhaust.

Biological particles and paper fibers together represented 3 to 19% (2-15 ug/m3) of the PM10. Biological particle types consisted of spores, conidia, hyphae, plant tissue fragments, plant hairs and insect parts.

Glassy flyash spheres were detected in low concentrations in the samples, and indicated impact from coal combustion source emissions in the sampling area. Flyash concentrations represented less than 1% of the PM10 in each of the samples.

Chemical Analysis Samples—

Sulfates represented 30% and 19% (20 and 13 ug/m3) of the mass on the two samples submitted for chemical analysis only. If the sulfates occur as the simple ammonium salt, then this component represents 41% and 25% (27 and 18 ug/m3) of the PM10. Nitrate concentrations, calculated as the simple ammonium salt, comprised less than 1% (<1 ug/m3) of the PM10 in both samples.

2. SAMPLES SUBMITTED

Six PM10 samples collected on 8" x 10" quartz fiber filters were submitted to IIT Research Institute (IITRI) for analysis. Four of the samples were submitted for full PM10 analysis and two samples were submitted for chemical analysis only. The objective of the full analysis was to determine the coal contribution to the PM10. Two samples were submitted for chemical analysis to determine the sulfate and nitrate contributions to the PM10.

Table 2 lists the sample identification information provided.

Table 2. SAMPLE IDENTIFICATION INFORMATION

Filter No.	Sampling Date	Sampling Site	Total Mass, g	PM10, ug/m3
Full Analysis				
12	6/15/89	Q180JA	0.131	80.6
55	8/04/89	Q180JA	0.186	116.7
62	8/14/89	Q180JA	0.032	20.1
45	7/27/89	Q180JA	0.095	58.4
Chemical Analysis				
23	6/27/89	Q180JB	0.116	65.9
54	8/05/89	Q180JB	0.124	71.8

3. ANALYSIS METHODS

3.1 ANALYTICAL OVERVIEW

Polarized light microscopy, low temperature ashing, particle size analysis, and ion chromatography were the analytical methods selected to identify and quantify the sample components on the full analysis samples. Polarized light microscopy (PLM) was used to identify the collected aerosols and to provide a semi-quantitative estimation of some sample constituent concentrations. Low temperature ashing (LTA) provided a quantitative measure of the combined weight associated with organic compounds and elemental carbon. Particle size analysis was used to determine the individual concentrations of the coal, other carbon and biological particles that were measured together as the weight lost in low temperature ashing. Ion chromatography (IC) was used on both the full analysis samples as well as the chemical analysis samples to quantify sulfate and nitrate ion concentrations. On the basis of the PLM phase identifications, the appropriate sulfate and nitrate salt concentrations were calculated from the ion concentrations.

Upon receipt, the filters were inspected for uniformity and any abnormalities such as water stains or tears. The whole filters were desiccated for 24 hours and then weighed prior to cutting for low temperature ashing and ion chromatography analyses. The total filter masses measured at IITRI were compared to the final weights recorded on the filter envelopes to determine if any significant weight change had occurred since the filters were collected.

3.2 LOW TEMPERATURE ASHING (LTA) ANALYSIS

A 1" x 10" strip was cut from each full analysis filter for low temperature ashing analysis. Each strip was weighed and then ashed in a radio frequency generated oxygen plasma asher. Ashing time for complete removal of combustible sample components was three hours at 475 watts. Upon removal from the asher, each sample strip was redesiccated and weighed to determine the mass lost in ashing.

From the mass of particles known to be present on the total filter, the mass of particles present on an ashed section was calculated by assuming that the total mass of particles was evenly distributed over the filter's effective collection area (7" x 9"). The mass percentage of the PM10 lost in the ashing process (LTA loss) was calculated by dividing the measured mass loss by the calculated mass of total particles on the ashed section.

3.3 ION CHROMATOGRAPHIC (IC) ANALYSES FOR SULFATE AND NITRATE

A 1" x 10" strip was cut from each filter and extracted with distilled, deionized water to dissolve water-soluble salts. The resulting solutions were filtered to remove particles and the remains of the quartz fiber filters. The ion concentrations in the extracts were determined with the ion chromatograph after dilution to known volumes.

The areas under the sulfate and nitrate peaks in the anion chromatograms generated were measured and converted to ion concentrations in the water extracts from standard calibration curves. The raw data were then multiplied by the extract volumes to yield the masses of ions per filter strip analyzed. Mass concentrations of sulfate and nitrate in the PM10 were then calculated from the mass of ions determined to be present on the filter section divided by the mass of total particles calculated to be present on the filter section.

3.4 POLARIZED LIGHT MICROSCOPY (PLM)

3.4.1 Sample Preparation

Sections of the unashed and ashed (full analysis) filter samples were mounted on glass slides in immersion oil (nD = 1.452) under coverslips. The immersion oil renders the quartz fibers invisible, thereby allowing transmission of light and observation of particle types collected on the filter surface and throughout the filter depth.

3.4.2 Particle Identification

The samples were analyzed with a Leitz optical microscope equipped for polarized light microscopy (PLM). Optical and physical properties of the particles were observed in order to identify the particle types.

The ashed and unashed sections of the filters mounted for PLM analysis were systematically scanned. Measurements of the largest (linear dimension) particle and estimated mean size for each identified particle type representing 1% or more of the sample mass were recorded on an individual microscopical sample analysis report form during this systematic scan.

The identified particle types are listed on the appended report forms as coal, other carbon (vehicle exhaust, oil soot and rubber tire fragments), biologicals (which also included some paper fibers), ammonium nitrate, ammonium sulfate, minerals (particles originating from pavement, gravel and soil components that are suspended by traffic, wind, construction, agriculture, etc., as well as materials suspended by bulk mineral handling or transport activities), and glassy flyash.

3.4.3 Particle Concentration Measurements

3.4.3.1 LTA Loss (Ashable) Components

The polarizing microscope was used to distinguish, size and count the ashable components in each full analysis sample. The ashable particle types were grouped into three categories for concentration determination by particle size analysis: coal, other carbon (vehicle exhaust, oil soot and rubber tire fragments), and biologicals (including paper fibers). These components (plus the nitrates, which were quantified by ion chromatography) comprise the measured LTA loss of the samples. These components were sized using the as-received (unashed) filter sections. From the particle sizes measured and numbers of particles of each size and type counted, the volumes of each particle type per unit area of filter were calculated. The volumes were then multiplied by appropriate density factors (1.21 g/cm3 for coal), to yield the masses of each ashable particle type per unit area of filter. The calculated particle type masses were normalized with the LTA loss data, after correction for the particle volume occupied by unashable coal residues and the ammonium nitrate concentrations as determined by IC.

A stratified counting approach was used in the particle size analysis. In stratified counting, several different magnifications are used to allow observation and identification of the wide range of particle sizes present. The small particles, which are generally the most abundant, are sized and counted at the higher magnifications. The larger particles, which generally are not abundant, are counted at low magnification, so that a large enough area of the filter can be viewed.

Particles were sized and counted with the aid of a British Standard Graticule (BSG). The BSG consists of two components: a field-of-view-defining rectangle; and a series of seven, graduated-diameter circles. Particles are sized in a field of view by comparing the projected area of the particle to the projected areas of the circles and selecting the circle that most closely corresponds to the particle's projected area. Each circle is 1.414 (the square root of 2) times larger than the previous circle; therefore, the circle areas increase by a factor of two.

The BSG field-of-view-defining rectangle is subdivided into seven smaller rectangles (field sizes). The rectangles decrease in area by a factor of two. The BSG, with its various field sizes, is ideally suited for use with the stratified counting approach because it provides a means for minimizing the particle counts that are made and it provides a simple basis for normalizing all of the counting data from the various magnifications. For example, the smallest particles are usually the most numerous and can amount to several thousand particles in the full (largest) rectangle. The stratified counting approach with the BSG allows the selection of a smaller rectangle size so that a greater number of fields of view can be counted before the predetermined statistically significant count value is reached. The larger particles are less abundant than the sub-2.5 um particles, so a larger rectangle is used for counting. In fact, to cover the full size range of less than 2.5 um to greater than 20 um, three different objective magnifications were used, along with appropriate field sizes.

The counting criteria established at the start of the analysis were designed to provide statistically significant counts for each particle type in each size range, allow examination of a representative percentage of the filter area and to be time and cost effective. Ideally, magnifications and field sizes were selected for each particle type to yield a total count of 30 particles in each size range over 20 fields of view. Counting was continued over more fields of view until the 30 counts were reached or until 100 fields of view (in the largest box size) had been examined. If 30 counts were recorded before 20 fields of view were examined, counting was continued until the minimum 20 fields of view were examined.

Error estimates for the component concentrations determined on the basis of the particle size data were calculated from the standard counting errors for each particle type in each particle size range.

3.4.3.2 LTA Residue (Non-ashable) Components

The non-ashable sample components were less than 50% of the PM10 mass in all four of the full analysis samples. In three of the four samples, ammonium sulfate more than 50% of the LTA residue. In the fourth sample, minerals were observed to be the dominant type of particle in the LTA residue. Therefore, the more rigorous particle size analysis determination of component concentrations in the LTA residue was not conducted.

Sulfate (and nitrate) concentrations were measured quantitatively by ion chromatography. For the full analysis samples, the sulfate was computed as ammonium sulfate and the nitrate concentrations were computed as ammonium nitrate. Recovery studies (by IITRI) show that the nitrate is lost during low temperature ashing while the sulfate is not. Therefore, the sulfate concentrations as ammonium sulfate that occur as part of the LTA residue were subtracted from the LTA residue mass. (The nitrate concentration as ammonium nitrate was subtracted from the LTA mass loss before normalizing the particle size data of the ashable components to the LTA loss values.) These adjusted LTA residue masses were then used to calculate the concentrations of the other non-ashable components in the LTA residues.

The concentrations of the individual components, or of several components grouped together, were microscopically estimated by their areal concentrations relative to the other particles in the LTA residue. The estimates were recorded as areal percentages. The LTA residue consisted predominantly of non-ashable particles such as minerals, flyash and sulfates. The areal concentrations of components in the LTA residue were normalized to the adjusted LTA residue masses. The final percentages of each component were calculated from the gravimetric values obtained from the low temperature ashing data. The reproducibility of the microscopical estimates, based on previous IITRI studies, are as follows:

Component Concentration	Uncertainty
25% - 100%	+/-20%
5% - 25%	+/-40%
0.5% - 5%	+/-100%
<0.5%	+/-200%

4. ANALYSIS RESULTS

4.1 MICROSCOPICAL ANALYSIS RESULTS

The individual microscopical sample analysis reports are contained in Appendix A. The data from these reports were summarized in Table 1.

4.2 LOW TEMPERATURE ASHING AND CHEMICAL ANALYSIS RESULTS

The results of the ion chromatographic analysis for sulfates and nitrates for all six samples, as well as the low temperature ashing analysis results for the four full analysis samples, are listed in Table 3. Measured LTA losses for filter numbers 62 and 45 exceeded 100%, probably due primarily to filter fiber loss during transport or handling of the filters. Therefore, LTA losses for these two filters were microscopically estimated.

4.3 COAL PARTICLE SIZE DISTRIBUTION

The coal particle size distributions by mass % and ug/m3 are listed in Table 4.

Respectfully submitted,

IIT RESEARCH INSTITUTE

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Principal Analyst
Fine Particles Research Group

Reviewed by,

Jean Graf-Teterycz

Jean Graf-Teterycz
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Table 3. RESULTS OF LOW TEMPERATURE ASHING AND CHEMICAL ANALYSIS

Filter No.	PM10, ug/m3	LTA Loss		Sulfate		Nitrate	
		%	ug/m3	%	ug/m3	%	ug/m3
12	80.6	51.9	41.8	6.1	5.0	0.5	0.4
55	116.7	67.7	79.0	19.9	23.3	0.1	0.1
62	20.1	83*	17*	11.1	2.2	2.9	0.1
45	58.4	60*	35*	26.7	15.6	0.1	0.1
23	65.9	NA	NA	30.1	19.8	0.1	0.1
54	71.8	NA	NA	18.5	13.3	0.1	0.1

*values estimated

Table 4. SUMMARY OF PARTICLE SIZE ANALYSIS RESULTS

SAMPLE IDENTIFICATION	12	55	62	45
SAMPLE SITE	Q180JA	Q180JA	Q180JA	Q180JA
SAMPLE DATE	6/15/89	8/4/89	8/14/89	7/27/89
AEROSOL CONC., ug/m3	80.6	116.7	20.1	58.4
IITRI NUMBER	C08025-001	C08025-002	C08025-003	C08025-006
MASS % LTA COAL	45.17	40.43	17.22	24.75
MASS % TOTAL COAL	49.39	44.21	18.83	27.06
MASS % OTHER CARBON	2.93	14.19	51.67	16.11
MASS % BIOLOGICALS	3.04	13.04	10.35	19.00
ug/m3 LTA COAL	36.41	47.18	3.46	14.45
est err LTA COAL, +/-ug/m3	2.47	4.01	0.49	1.81
ug/m3 TOTAL COAL	39.81	51.60	3.78	15.80
est err TOT COAL, +/-ug/m3	4.18	6.02	0.81	3.13
ug/m3 OTHER CARBON	2.36	16.56	10.39	9.41
est err OTH CARB, +/-ug/m3	0.12	0.62	0.54	0.56
ug/m3 BIOLOGICALS	2.45	15.22	2.08	11.10
est err BIOLOGS, +/-ug/m3	0.23	1.29	0.48	1.43
COAL MASS % SIZE DISTRIBUTION				
<2.5 um	1.27	2.59	7.91	2.96
2.5-5.0 um	20.10	22.81	27.75	28.66
5.0-7.8 um	26.51	23.06	19.91	22.22
7.8-10.0 um	26.71	30.53	25.30	22.21
10.0-20.0 um	16.34	13.36	11.12	15.00
>20.0 um	9.06	7.65	8.01	8.94
COAL SIZE DISTRIBUTION AS ug/m3				
<2.5 um	0.51	1.34	0.30	0.47
2.5-5.0 um	8.00	11.77	1.05	4.53
5.0-7.8 um	10.55	11.90	0.75	3.51
7.8-10.0 um	10.63	15.75	0.96	3.51
10.0-20.0 um	6.51	6.89	0.42	2.37
>20.0 um	3.61	3.94	0.30	1.41

Appendix A

INDIVIDUAL SAMPLE ANALYSIS REPORTS

PROJECT	C08025-001	FILTER NO.	12
AGENCY	Simpson Weather	PM10, ug/m3	80.6
MICROSCOPIST	E. Segers	SAMPLING SITE	Q180JA
REPORT DATE	9/27/89	SAMPLING DATE	6/15/89

51.9 % LTA LOSS 6.1 % SO4= 0.5 % NO3-

COMPONENT	CONCENTRATION (WEIGHT %)	GEOMETRIC SIZE, um MEAN	RANGE
Coal	49	8	0.5-96
Other carbon	3	<1	0.3-45
Biologicals, paper fibers	3	8	3-170
Ammonium nitrate	1	—	—
Ammonium sulfate	8	—	—
Minerals			
quartz, feldspars	6	6	0.2-56
carbonates	<1	—	0.2-11
clay, humus	9	1	0.5-23
other minerals (mica)	20	3	1-34
Glassy flyash	<1	—	0.2-9

PROJECT	C08025-002	FILTER NO.	55
AGENCY	Simpson Weather	PM10, ug/m3	116.7
MICROSCOPIST	E. Segers	SAMPLING SITE	Q180JA
REPORT DATE	9/27/89	SAMPLING DATE	8/04/89

67.7 % LTA LOSS

19.9 % SO4=

0.1 % NO3-

COMPONENT	CONCENTRATION (WEIGHT %)	GEOMETRIC SIZE, um MEAN	RANGE
Coal	44	8	0.5-93
Other carbon	14	<1	0.3-23
Biologicals, paper fibers	13	8	3-192
Ammonium nitrate	<1	—	—
Ammonium sulfate	27	—	—
Minerals			
quartz, feldspars	<1	—	—
carbonates	<1	—	—
clay, humus	<1	—	—
other minerals (mica)	1	3	1-39
Glassy flyash	<1	—	0.2-5

PROJECT	C08025-003	FILTER NO.	62
AGENCY	Simpson Weather	PM10, ug/m3	20.1
MICROSCOPIST	E. Segers	SAMPLING SITE	Q180JA
REPORT DATE	9/27/89	SAMPLING DATE	8/14/89

83* % LTA LOSS

11.1 % SO4=

2.9 % NO3-

COMPONENT	CONCENTRATION (WEIGHT %)	GEOMETRIC SIZE, um MEAN	RANGE
Coal	19	8	0.5-93
Other carbon	52	<1	0.3-23
Biologicals, paper fibers	10	8	3-192
Ammonium nitrate	4	—	—
Ammonium sulfate	15	—	—
Minerals			
quartz, feldspars	<1	—	—
carbonates	<1	—	—
clay, humus	<1	—	—
other minerals (mica)	<1	3	1-39
Glassy flyash	<1	—	0.2-5

*Estimated value

PROJECT	C08025-004	FILTER NO.	45
AGENCY	Simpson Weather	PM10, ug/m3	58.4
MICROSCOPIST	E. Segers	SAMPLING SITE	Q180JA
REPORT DATE	9/27/89	SAMPLING DATE	7/27/89

60* % LTA LOSS   26.7 % SO4=   0.1 % NO3-

COMPONENT	CONCENTRATION (WEIGHT %)	GEOMETRIC SIZE, um MEAN	RANGE
Coal	27	8	0.5-62
Other carbon	16	<1	0.3-40
Biologicals, paper fibers	19	5	3-170
Ammonium nitrate	<1	—	—
Ammonium sulfate	37	—	—
Minerals			
quartz, feldspars	<1	—	—
carbonates	<1	—	—
clay, humus	<1	—	—
other minerals (mica)	<1	—	—
Glassy flyash	<1	—	—

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5 October 1989

Simpson Weather Associates, Inc.
809 East Jefferson St.
Charlottesville, VA 22902

Attention: Mr. G.D. Emmitt

Subject: Microscopical and Chemical Analysis of Six PM10 Samples
Letter Report on IITRI Project No. C08025

Dear Mr. Emmitt:

Six samples of airborne particulate less than 10 um in aerodynamic diameter (PM10) were submitted by Simpson Weather Associates for analysis. The samples were collected near the Dominion Terminal Associates' coal storage facility in Newport News, VA. Four of the samples were submitted for full PM10 analysis (microscopical, chemical and low temperature ashing analysis). The primary goal of the full analyses was to determine the concentration of coal particles in the PM10. Two samples were submitted only for chemical analysis to determine the contributions made by sulfate and nitrate salts to the PM10. None of the samples represented an exceedance of the USEPA primary air quality standard for PMIO of 150 ug/m3.

1. SUMMARY OF RESULTS

Full Analysis Samples-

Raw coal represented 49% (40 ug/m3), 44% (52 ug/m3) and 27% (16 ug/m3) of the sample mass in the three full analysis samples with the highest PM10 levels, and 19% (4 ug/m3) of the sample mass in the low PMIO concentration sample (see Table 1). The mean particle size of the coal in each of the four samples was 8 um.

Ammonium sulfate comprised 8% (7 ug/m3). 27% (32 ug/m3) and 37% (21 ug/m3) of the PM10 in the higher level samples, and 15% (3 ug/m3) of the PM10 in the low level sample. Ammonium nitrate occurred in relatively low concentrations, representing up to 1% (<I-l ug/m3) of the PM10 in the higher level samples and 4% (1 ug/m3) of the PM1O in the low level sample.

Minerals were a significant component of only one of the four samples, contributing 35% (28 ug/m3) of the PM10 in one of the higher level samples, and up to 1% (4-1 ug/m3) of the aerosol in the other three samples. Soil-derived mineral types, principally mica, clay and quartz, were predominant.

Particles identified as "other carbon" in the samples consisted of vehicle exhaust from gasoline and diesel engines, oil soot and rubber tire fragments.

Table 1. SUMMARY OF MICROSCOPICAL ANALYSIS RESULTS

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Ammonium nitrate	1	1	<1	<1	4	1	<1	<1
Minerals	35	28	1	1	<1	<1	<1	<1
Other Carbon	3	2	14	17	52	10	16	9
Biologicals, paper	3	2	13	15	10	2	19	11
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These components together represented 3%, (2 ug/m3), 14% (17 ug/m3) and 16% (9 ug/m3) of the aerosol in the higher concentration PM10 samples, and 52% (10 ug/m3) of the aerosol in the low concentration PM10 sample. The majority of the if other carbon" was comprised of vehicle exhaust.

Biological particles and paper fibers together represented 3 to 19% (2-15 ug/m3) of the PM10. Biological particle types consisted of spores, conidia, hyphae, plant tissue fragments, plant hairs and insect parts.

Glassy flyash spheres were detected in low concentrations in the samples, and indicated impact from coal combustion source emissions in the sampling area. Flyash concentrations represented less than 1% of the PM10 in each of the samples.

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From the mass of particles known to be present on the total filter, the mass of particles present on an ashed section was calculated by assuming that the total mass of particles was evenly distributed over the filter's effective collection area (7" X 911). The mass percentage of the PM10 lost in the ashing process (LTA loss) was calculated by dividing the measured mass loss by the calculated mass of total particles on the ashed section.

3.3 ION CHROMATOGRAPHIC (IC) ANALYSES FOR SULFATE AND NITRATE

A I" x 10" strip was cut from each filter and extracted with distilled, deionized water to dissolve water-soluble salts. The resulting solutions were filtered to remove particles and the remains of the quartz fiber filters. The ion concentrations in the extracts were determined with the ion chromatograph after dilution to known volumes.

The areas under the sulfate and nitrate peaks in the anion chromatograms

generated were measured and converted to ion concentrations in the water extracts from standard calibration curves. The raw data were then multiplied by the extract volumes to yield the masses of ions per filter strip analyzed. Mass concentrations of sulfate and nitrate in the PM10 were then calculated from the mass of ions determined to be present on the filter section divided by the mass of total particles calculated to be present on the filter section.

3.4 POLARIZED LIGHT MICROSCOPY (PLM)

3.4.1 Sample Preparation

Sections of the unashed and ashed (full analysis) filter samples were mounted on glass slides in immersion oil (nD = 1.452) under coverslips. The immersion oil renders the quartz fibers invisible, thereby allowing transmission of light and observation of particle types collected on the filter surface and throughout the filter depth.

3.4.2 Particle Identification

The samples were analyzed with a Leitz optical microscope equipped for polarized light microscopy (PLM). Optical and physical properties of the particles were observed in order to identify the particle types.

The ashed and unashed sections of the filters mounted for PLM analysis were systematically scanned. Measurements of the largest (linear dimension),rrticle and estimated mean size for each identified particle type representing or more of the sample mass were recorded on an individual microscopical sample analysis report form during this systematic scan.

The identified particle types are listed on the appended report forms as coal, other carbon (vehicle exhaust, oil soot and rubber tire fragments), biologicals (which also included some paper fibers), ammonium nitrate, ammonium sulfate, minerals (particles originating from pavement, gravel and soil components that are suspended by traffic, wind, construction, agriculture, etc., as well as materials suspended by bulk mineral handling or transport activities), and glassy flyash.

3.4.3 Particle Concentration Measurements

3.4.3.1 LTA Loss (Ashable) Components

The polarizing microscope was used to distinguish, size and count the ashable components in each full analysis sample. The ashable particle types were grouped into three categories for concentration determination by particle size analysis: coal, other carbon (vehicle exhaust, oil soot and rubber tire fragments), and biologicals (including paper fibers). These components (plus the nitrates, which were quantified by ion chromatography) comprise the measured LTA loss of the samples. These components were sized using the as-received (unashed) filter sections. From the particle sizes measured and numbers of particles of each size and type counted, the volumes of each particle type per unit area of filter were calculated. The volumes were then multiplied by appropriate density factors (1.21 g/cm3 for coal),.to yield the masses of each

ashable particle type per unit area of filter. The calculated particle type masses were normalized with the LTA loss data, after correction for the particle volume occupied by unashable coal residues and the ammonium nitrate concentrations as determined by IC.

A stratified counting approach was used in the particle size analysis. In stratified counting, several different magnifications are used to allow observation and identification of the wide range of particle sizes present. The small particles, which are generally the most abundant, are sized and counted at the higher magnifications. The larger particles, which generally are not abundant, are counted at low magnification, so that a large enough area of the filter can be viewed.

Particles were sized and counted with the aid of a British Standard Graticule (BSG). The BSG consists of two components: a field-of-view-defining rectangle; and a series of seven, graduated-diameter circles. Particles are sized in a field of view by comparing the projected area of the particle to the projected areas of the circles and selecting the circle that most closely corresponds to the particle's projected area. Each circle is 1.414 (the square root of 2) times larger than the previous circle; therefore, the circle areas increase by a factor of two.

The BSG field-of-view-defining rectangle is subdivided into seven smaller rectangles (field sizes). The rectangles decrease in area by a factor of two.

The BSG, with its various field sizes, is ideally suited for use with the stratified counting approach because it provides a means for minimizing the particle counts that are made and it provides a simple basis for normalizing all of the counting data from the various magnifications. For example, the smallest particles are usually the most numerous and can amount to several thousand particles in the full (largest) rectangle. The stratified counting approach with the BSG allows the selection of a smaller rectangle size so that a greater number of fields of view can be counted before the predetermined statistically significant count value is reached. The larger particles are less abundant than the sub-2.5 um particles, so a larger rectangle is used for counting. In fact, to cover the full size range of less than 2.5 um to greater than 20 um, three different objective magnifications were used, along with appropriate field sizes.

The counting criteria established at the start of the analysis were designed to provide statistically significant counts for each particle type in each size range, allow examination of a representative percentage of the filter area and to be time and cost effective. Ideally, magnifications and field sizes were selected for each particle type to yield a total count of 30 particles in each size range over 20 fields of view. Counting was continued over more fields of view until the 30 counts were reached or until 100 fields of view (in the largest box size) had been examined. If 30 counts were recorded before 20 fields of view were examined, counting was continued until the minimum 20 fields of view were examined.

Error estimates for the component concentrations determined on the basis of the particle size data were calculated from the standard counting errors for each particle type in each particle size range.

3.4.3.2 LTA Residue (Non-ashable) Components

The non-ashable sample components were less than 50% of the PM10 mass in all four of the full analysis samples. In three of the four samples, ammonium sulfate more than 50% of the LTA residue. In the fourth sample, minerals were observed to be the dominant type of particle in the LTA residue. Therefore, the more rigorous particle size analysis determination of component concentrations in the LTA residue was not conducted.

Sulfate (and nitrate) concentrations were measured quantitatively by ion chromatography. For the full analysis samples, the sulfate was computed as ammonium sulfate and the nitrate concentrations were computed as ammonium nitrate. Recovery studies (by IITRI) show that the nitrate is lost during low temperature ashing while the sulfate is not. Therefore, the sulfate concentrations as ammonium sulfate that occur as part of the LTA residue were subtracted from the LTA residue mass. (The nitrate concentration as ammonium nitrate was subtracted from the LTA mass loss before normalizing the particle size data of the ashable components to the LTA loss values.) These adjusted LTA residue masses were then used to calculate the concentrations of the other non-ashable components in the LTA residues.

The concentrations of the individual components, or of several components grouped together, were microscopically estimated by their areal concentrations relative to the other particles in the LTA residue. The estimates were recorded as areal percentages. The LTA residue consisted predominantly of non-ashable particles such as minerals, flyash and sulfates. The areal concentrations of components in the LTA residue were normalized to the adjusted LTA residue masses. The final percentages of each component were calculated from the gravimetric values obtained from the low temperature ashing data. The reproducibility of the microscopical estimates, based on previous IITRI studies, are as follows:

Component Concentration	Uncertainty
25% - 100%	+/-20%
5% - 25%	+/-40%
0.5% - 5%	+/-100%
<0.5%	+1-200%

4. ANALYSIS RESULTS

4.1 MICROSCOPICAL ANALYSIS RESULTS

The individual microscopical sample analysis reports are contained in Appendix A. The data from these reports were summarized in Table 1.

4.2 LOW TEMPERATURE ASHING AND CHEMICAL ANALYSIS RESULTS

The results of the ion chromatographic analysis for sulfates and nitrates for all six samples, as well as the low temperature ashing analysis results for the four full analysis samples, are listed in Table 3. Measured LTA losses for filter numbers 62 and 45 exceeded 100%, probably due primarily to filter fiber loss during transport or handling of the filters. Therefore, LTA losses for these two filters were microscopically estimated.

4.3 COAL PARTICLE SIZE DISTRIBUTION

The coal particle size distributions by mass Z and ug/m3 are listed in Table 4.

Respectfully submitted,

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Reviewed by,

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Table 3. RESULTS OF LOW TEMPERATURE ASHING AND CHEMICAL ANALYSIS

	PM10,	LTA Loss		Sulfate		Nitrate	
Filter No.	ug/m3	%	ug/m3	%	ug/m3	% -	ug/m3
12	80.6	51.9	41.8	6.1	5.0	0.5	0.4
55	116.7	67.7	79.0	19.9	23.3	0.1	0.1
62	20.1	83*	17*	11.1	2.2	2.9	0.1
45	58.4	60*	35*	26.7	15.6	0.1	0.1
23	65.9	NA	NA	30.1	19.8	0.1	0.1
54	71.8	NA	NA	18.5	13.3	0.1	0.1

*values estimated

Table 4. SUMMARY OF PARTICLE SIZE ANALYSIS RESULTS

SAMPLE IDENTIFICATION 12 55 62 45
SAMPLE SITE Q180JA Q180JA Q180JA Q180JA
SAMPLE DATE 6/15/89 8/4/89 8/14/89 7/27/89
AEROSOL CONC., ug/m3 80.6 116.7 20.1 58.4
IITRI NUMBER C08025-001 C08025-002 C08025-003 C08025-006

MASS % LTA COAL 45.17 40.43 17.22' 24.75
MASS % TOTAL COAL 49.39 44.21 18.83 27.06
MASS % OTHER CARBON 2.93 14.19 51.67 16.11
MASS % BIOLOGICALS 3.04 13.04 10.35 19.00

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uq/m3 LTA COAL 36.41 47.18 3.46 14.45
est err LTA COAL, +/-L49/Tfiq- 2.47 4.01 0.49 1.31

ug/m3 TOTAL COAL 39.81 51.60 3.78 15.80
est err TOT COAL, + @3 4.18 6. (12 0.81 3.13

ug/m3 OTHER CARBON 2.36 16.56 10.39 9.41
est err OTH CARB, +/-LJ9/m3 0. 12 0.62 0.54 0.56

Ug/m3 BIOLOGICALS 2.45 15.22 2.08 11.10
est err BIOLOGS, +/-L49/MZ4 0.23 1.29 0.48 1.43

COAL MASS % SIZE DISTRIBUTION
C-2-5 LIM 42. 59 7.91 2.96
2.5-5.0 UM 20. 1 0 22.81 27.75 28.66
5.0-7.3 LIM 26.51 23.06 19.91 22.22
7.8-10.0 LIM 26.71 30.53 25.30 22.21
10.0-20-0 LIM 16. 34 1 13 - 36 11.12 15.00
>20.0 um 19.06 7.65 8.01 8.94

COAL SIZE DISTRIBUTION AS ug/m3
1-34 0.30
,.2- 5 LIM 0.51 0.47
2.5-5.0 um 8.00 11.77 1.05 4.53
5.0-7.8 LIM 10.55 11.90 0.75 3.51
7.8-10.0 LIM 10.63 15.75 0.96 3.51
1 0. 0 -20. 0 LIM 6.51 6-89 0.42 2.37
>20. 0 um 3.61 3.94 0.30 1.41

Appendix A
INDIVIDUAL SAMPLE ANALYSIS REPORTS

PROJECT C08025-001 FILTER NO. 12
AGENCY Simpson Weather PM10, ug/m3 90. 6
MICROSCOPIST E. Seqers SAMPLING SITE -Q180JA
REPORT DATE -9/27/89 SAMPLING DATE 6/15189

51.9 % LTA LOSS 6.1 % S04= 0.5 % N03-

CONCENTRATION GEOMETRIC SIZE, um
COMPONENT (WEIGHT %) MEAN RANGE

Coal 49 8 0.5-96

Other carbon 3 <1 0.3-45

Biologicals, paper fibers 3 8 3-170

Ammonium nitrate I

Ammonium sulfate 8

Minerals
quartz, feldspars 6 6 0.2-56
carbonates <1 0.2-11
clay, humus 9 1 0.5-23
other minerals (mica) 20 3 1-34

Glassy flyash <1 0.2-9

PROJECT C08025-002 FILTER NO. 55
AGENCY Simeson Weather PM10, ug/M3 116.7
MICROSCOPIST E. Segers SAMPLING SITE Q180JK-
REPORT DATE 9127/89 SAMPLING DATE 8/04/89

67.7 % LTA LOSS 19.9 % S04-- 0.1 % N03-

CONCENTRATION GEOMETRIC SIZE, um
COMPONENT (WEIGHT %) MEAN RANGE

Coal 44 8 0.5-93

Other carbon 14 <1 0.3-23

Biologicals, paper fibers 13 8 3-192

Ammonium nitrate <1

Ammonium sulfate 27

Minerals
quartz, feldspars <1
carbonates <1
clay, humus <1
other minerals (mica) 1 3 1-39

Glassy flyash <1 0.2-5

PROJECT C08025-003 FILTER NO. 62
AGENCY Simpson Weather PMlO1 ug/mT_ 20.1
MICROSCOPIST E. Seqers -SAMPLING SITE 9180JA
REPORT DATE 9T27/89 SAMPLING DATE 8/14/89

83* % LTA LOSS 11.1-% S04-- 2.9 % N03-

CONCENTRATION GEOMETRIC SIZE, um
COMPONENT (WEIGHT %) MEAN RANGE

Coal 19 8 0.5-93

Other carbon 52 <1 0.3-23

Biologicals, paper fibers 10 8 3-192

Ammonium nitrate 4

Ammonium sulfate 15

Minerals
quartz, feldspars <1
carbonates <1
clay, humus <1
other minerals (mica) <1 3 1-39

Glassy flyash < 1 0.2-5

*Estimated value

PROJECT C08025-004 FILTER NO. 45
AGENCY -----.Simpson Weather PMlO' ug/M3 58.4
MICROSCOPIST E. Seqers SAMPLING SITE 01807-
REPORT DATE 9/27/89 SAMPLING DATE 7/27189

60* % LTA LOSS 26.7 % S04= 0.1 % N03-

CONCENTRATION GEOMETRIC SIZE, um
COMPONENT (WEIGHT %) MEAN RANGE

Coal 27 8 0.5-62

Other carbon 16 <1 0.3-40

Biologicals, paper fibers 19 5 3-170

Ammonium nitrate <1

Ammonium sulfate 37

Minerals
quartz, feldspars <1
carbonates <1
clay, humus <1
other minerals (mica) <1

Glassy flyash <1

*Estimated value