

## **PM<sub>2.5</sub> CHEMICAL SPECIATION RESULTS FOR TEXAS – IMPLICATIONS FOR REGIONAL STUDIES**

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### **ABSTRACT**

PM<sub>2.5</sub> chemical speciation results were examined for a three-year period (2000 – 2002) from a network of 23 sites in Texas that are part of a national U.S. Environmental Protection Agency (EPA) PM<sub>2.5</sub> chemical speciation monitoring program. Three sites are part of EPA's Speciation Trends Network (STN), which uses EPA-designated samplers and an EPA contract laboratory, while the other 20 sites use the same laboratory but alternative samplers. Data were obtained from EPA's Air Quality System (AQS) and supplemented with information from the contractor laboratory. Initial results raised questions about the data in AQS — including no blank correction of reported results; potential overestimation of mass from the species reported; and lack of agreement for some basic species, such as total carbon and iron — compared to the results at the Interagency Monitoring for the Protection of Visual Environments (IMPROVE) Big Bend National Park site. A more rigorous analysis of the speciation data was conducted that included the use of blank corrected data, propagated measurement uncertainties, and an assessment of the differences between the corrected and uncorrected data and results. The analysis highlighted the need for careful planning and oversight of regional studies. This paper discusses the potential problems in such studies and provides recommendations to improve the quality and usefulness of PM<sub>2.5</sub> chemical speciation network results.

### **INTRODUCTION**

In 1997, the U.S. Environmental Protection Agency (EPA) promulgated new National Ambient Air Quality Standards (NAAQS) for particulate matter, including PM<sub>2.5</sub>. [1] In 1999, the first of roughly 1,500 Federal Reference Method (FRM) monitoring sites were established nationally (with more than 50 in Texas) to determine PM<sub>2.5</sub> mass concentrations and compliance with the PM<sub>2.5</sub> NAAQS. In 2000, EPA also established a smaller chemical speciation network of approximately 225 sites nationally (with 23 in Texas) to assess the chemical composition of PM<sub>2.5</sub>. The network was to help implement the PM<sub>2.5</sub> NAAQS by using the chemical speciation data to track the progress of controls, aid in interpreting health studies by linking effects to exposure to PM<sub>2.5</sub> constituents, and understand the effects of atmospheric constituents on visibility impairment. A subset of the speciation network, the Speciation Trends Network (STN), consists of about 54 sites nationally (with three in Texas) and provides an analysis of PM<sub>2.5</sub> composition trends nationally. The STN sites must use EPA-designated speciation samplers and the EPA contractor laboratory, Research Triangle

Institute (RTI). The other speciation sites can use alternate sampling methods and other laboratories, with EPA approval.

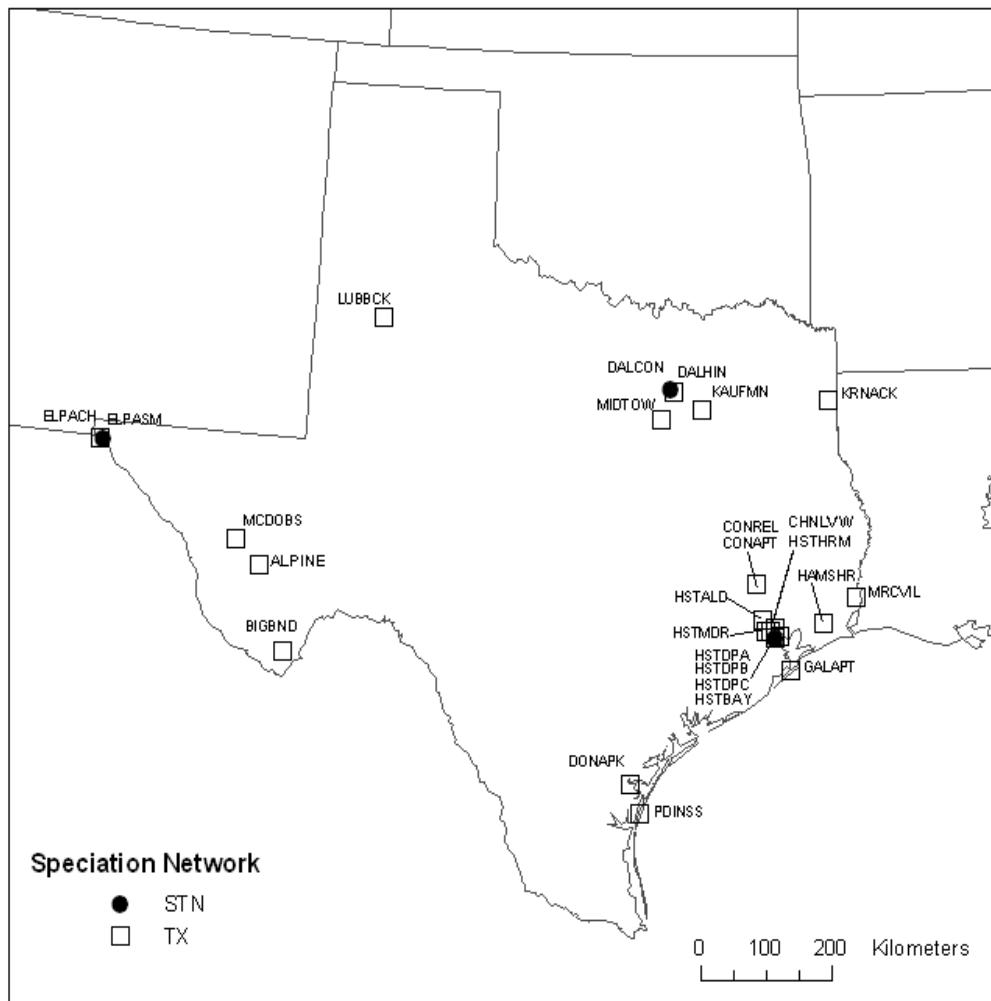
Initially, DRI examined the EPA Air Quality System (AQS) PM<sub>2.5</sub> chemical speciation data for Texas at the request of the Texas Commission on Environmental Quality (TCEQ) to provide updated quarterly summaries of chemical composition at each of the sites. However, a preliminary analysis raised concerns, including the lack of blank correction of the reported results, the potential overestimation of mass from the species reported, and the lack of agreement for some basic species markers such as total carbon and iron when compared to results at the IMPROVE Big Bend National Park site. [2] These concerns led to a more rigorous review [3-6] of the chemical speciation data. The review included an assessment of blank values; blank correction of sample data; level 2 validation check of the blank-corrected data; estimation of uncertainties through a propagation of error analysis; use of “corrected” data in temporal, spatial, and receptor analyses; and a comparison of differences in the results depending on whether the “uncorrected” or “corrected” data sets are used. Many of the findings have implications for the suitability of the Texas data and for the design and implementation of other large-scale monitoring efforts.

## **DESCRIPTION OF TEXAS NETWORK AND DATA**

Figure 1 shows the location of the PM<sub>2.5</sub> chemical speciation sites in Texas. Sites were phased in over the three-year period of 2000-2002. One site, Houston Regional Monitoring #3, was discontinued and another, Conroe, was relocated. Two sites, Lubbock and Padre Island National Seashore, began operation in 2003. Only one site, Houston Deer Park, had collocated speciation samplers, and no site permitted ongoing comparisons of STN and non-STN sampler results.

Three of the sites – Dallas Hinton, El Paso Chamizal, and Houston Deer Park – are STN sites, while the Deer Park site also has a collocated STN sampler. All the STN sites sample on a once-every-third-day sampling schedule and use the University Research Glassware (URG) Mass Aerosol Speciation Sampler (MASS), which is the EPA-designated speciation sampler used in Texas. The sampler has two units, each of which samples at a flow rate of 16.7 l/m<sup>3</sup>. The first unit has a PM<sub>10</sub> size selective inlet, followed by a magnesium oxide denuder (to absorb acidic gases), and a WINS impactor with a PM<sub>2.5</sub> size cut. After the impactor, a Teflon-membrane front filter is used for determination of mass by gravimetry, 48 elements by energy dispersive x-ray fluorescence (XRF), and ions (ammonium, potassium, sodium, sulfate, and non-volatile nitrate) by ion chromatography (IC). A backup nylon filter is used to determine volatile nitrate by IC and total nitrate as the sum of volatile nitrate and non-volatile nitrate. The second unit consists of a PM<sub>10</sub> size selective inlet, followed by a WINS impactor with a PM<sub>2.5</sub> size cut, and a quartz-fiber filter.

The quartz-fiber filter is used to determine organic, elemental, and total carbon using a thermal/optical transmittance (TOT) technique. The TOT carbon analysis method differs from the thermal/optical reflectance (TOR) method used by the Interagency Monitoring for the Protection of Visual Environments (IMPROVE) program. The two methods give different results for elemental and organic carbon fractions, although total carbon is expected to be similar. [7, 8]



**Figure 1. Texas PM<sub>2.5</sub> chemical speciation sampling sites.**

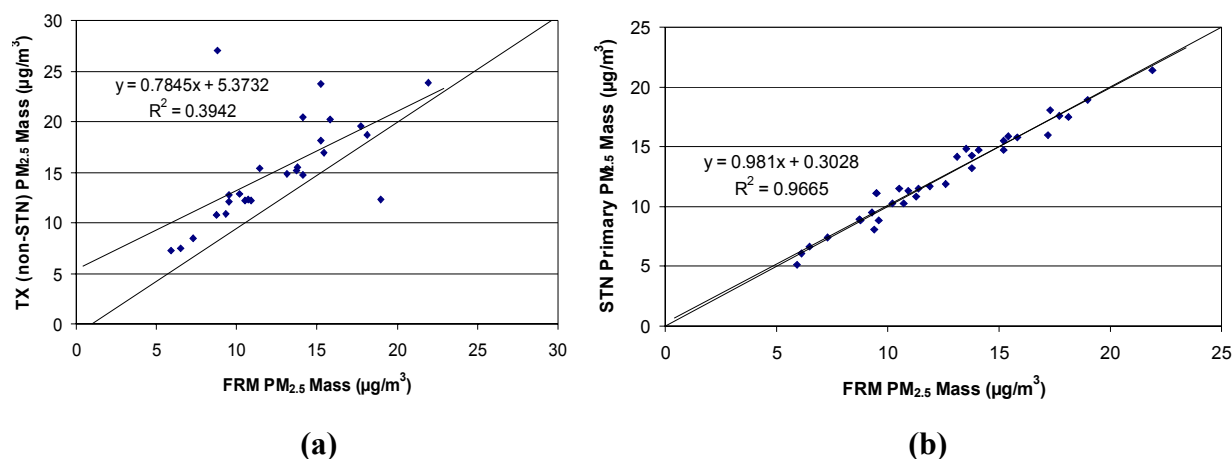
Twenty sites in Texas did not use EPA-designated speciation samplers. Half these sites sample on a one-in-six sampling frequency while the other half use a one-in-three frequency. During two summer periods (August 15 through September 30, 2000, and July 1 through August 5, 2001) seven sites in the greater Houston area sampled each day to support special studies.

Carbon analysis results by TOT were always obtained from quartz-fiber filters. However, the filter used for the analysis of ions by IC has changed over time. Prior to August 15, 2000, at the pilot site at Deer Park, ions by IC were obtained from Teflon-membrane filters. From August 15, 2000, through June 3, 2001, ions by IC were obtained from quartz-fiber filters at all non-STN sites. However, there were problems with high blank levels and almost all sodium ion results were voided for the period. Starting June 6, 2001, ions by IC were again obtained from Teflon-membrane filters.

There were more than 3,500 data sets for the Texas PM<sub>2.5</sub> chemical speciation network in AQS covering the three-year period 2000-2002. They included a complete data set defined as all parameters (up to 58 for STN samplers and 56 for non-STN samplers) measured for a given sampler set on the same date. However, many of the data sets are missing one or more of the major species (e.g., ions, elements or carbon). The data in AQS for Texas and elsewhere were also limited by what could be reported to AQS under an older reporting format (e.g., there are no blank values and reported values do not have associated Method Detection Limits [MDLs] or measurement uncertainties). Now, MDLs, uncertainties, and blank values are to be reported to AQS. However, none of the existing sample data are blank corrected. EPA has not indicated that speciation data will be blank corrected in the future or that it will provide a protocol to do so. As discussed below, this has significant consequences for determining the relative percent composition of the reconstructed mass and the potential contribution of certain sources to PM<sub>2.5</sub> mass concentrations.

## ASSESSMENT RESULTS AND DISCUSSION

The preliminary assessment utilized data that had not been blank corrected. At the Houston Deer Park site, a non-STN sampler operated for a brief period with an FRM sampler and an STN speciation sampler. However, as Figure 2 indicates, operational problems caused the non-STN sampler to have relatively poor agreement with the other two samplers for PM<sub>2.5</sub> mass. Thus, no further comparison was conducted for PM<sub>2.5</sub> constituents. The STN sampler, however, showed excellent agreement with the FRM sampler. Data for the collocated STN samplers at Houston Deer Park, as shown in Table 1, indicated reasonable precision for major constituents for 52 sets of paired data. The relative percent difference (RPD) was generally 12% or less and was worse for chlorine with 21%.

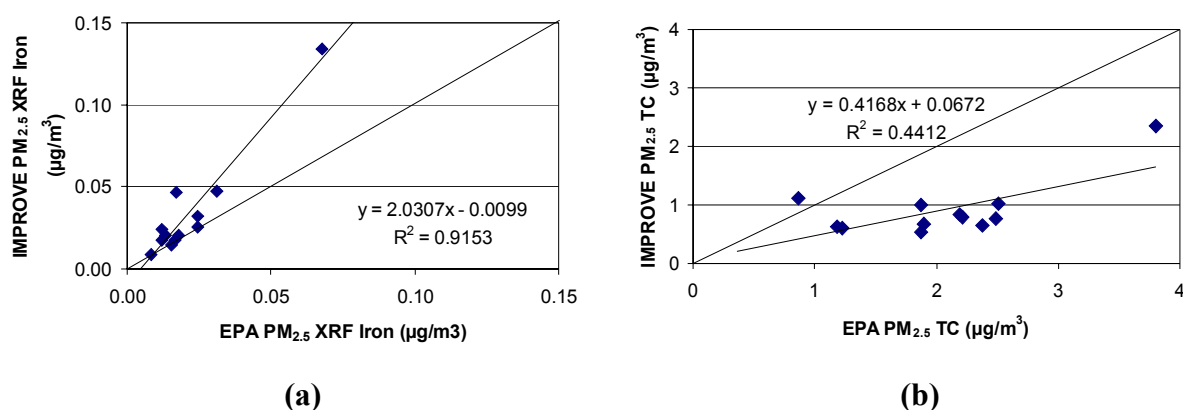


**Figure 2. Regression results for PM<sub>2.5</sub> mass for (a) STN and (b) non-STN sampler versus the FRM sampler at the Houston Deer Park site.**

PM <sub>2.5</sub> Species	Mean <sub>HSTDPB</sub> (µg/m <sup>3</sup> )	StdDev <sub>-HSTDPB</sub> (µg/m <sup>3</sup> )	Mean <sub>HSTDPC</sub> (µg/m <sup>3</sup> )	StdDev <sub>-HSTDPC</sub> (µg/m <sup>3</sup> )	RPD (%)
PM <sub>2.5</sub> Mass	10.60	4.69	9.74	4.30	8.53
Sulfate	3.33	1.93	2.99	1.72	10.58
Non-volatile Nitrate	0.38	0.44	0.32	0.44	-0.09
Ammonium	1.19	0.80	1.05	0.71	12.17
Sodium Ion	0.20	0.20	0.21	0.21	-3.68
Potassium Ion	0.05	0.03	0.06	0.03	8.50
Organic Carbon	2.69	1.44	2.54	1.48	5.62
Elemental Carbon	0.33	0.18	0.35	0.18	-5.30
Aluminum	0.04	0.08	0.04	0.09	-8.70
Barium	0.02	0.01	0.02	0.01	5.30
Calcium	0.04	0.03	0.05	0.03	-10.74
Chlorine	0.04	0.11	0.05	0.12	-20.76
Iron	0.05	0.06	0.06	0.06	-5.94
Magnesium	0.01	0.02	0.01	0.02	5.86
Potassium	0.07	0.04	0.06	0.04	4.47
Silicon	0.14	0.19	0.14	0.21	-6.51
Sodium	0.14	0.19	0.15	0.19	-2.43
Sulfur	1.03	0.58	0.94	0.53	9.12

**Table 1. Precision for major PM<sub>2.5</sub> constituents for STN samplers at Deer Park.**

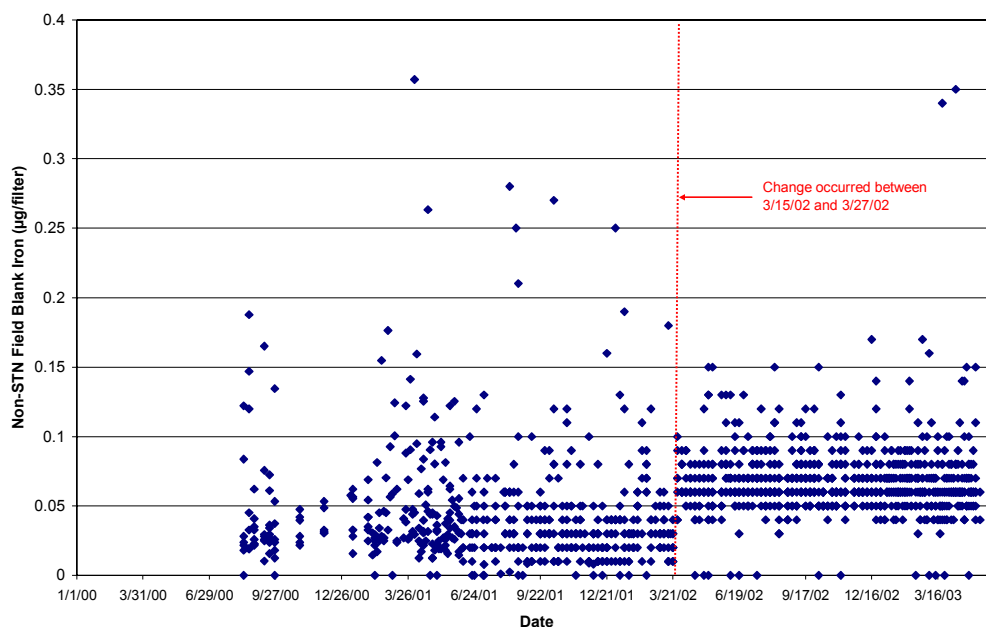
Because of operational problems with the non-STN sampler collocated with the IMPROVE sampler at Big Bend National Park, only 12 pairs of data sets could be used in the initial assessment. Figure 2 illustrates the apparent lack of agreement between the IMPROVE and EPA speciation results for iron and total carbon. However, excellent agreement was found for PM<sub>2.5</sub> mass and sulfate.



**Figure 3. IMPROVE versus EPA regression plots for (a) iron and (b) total carbon.**

RTI provided field and trip blank data for the expanded analysis. Examination of the blank data indicated that statistically significant differences for some species were obtained that varied by type of blank (e.g., field versus trip blank for non-STN samplers only), type of sampler (i.e., STN versus non-STN sampler), site, and type of filter (i.e., quartz-fiber versus Teflon-membrane for non-STN sampler ions). Thus, blank corrections were made by

subtracting the mean value (with values greater than three standard deviations from the mean removed) of the field blanks by type of sampler, site, year, and filter type. A review of field blank values over time indicated that some of the differences were likely related to changes in laboratory operations (e.g., analytical instrument used, filter lots, possible change in laboratory procedures, etc.). Figure 4 shows one such plot, for iron, with noticeable changes over time.



**Figure 4. Time series of non-STN field blank iron.**

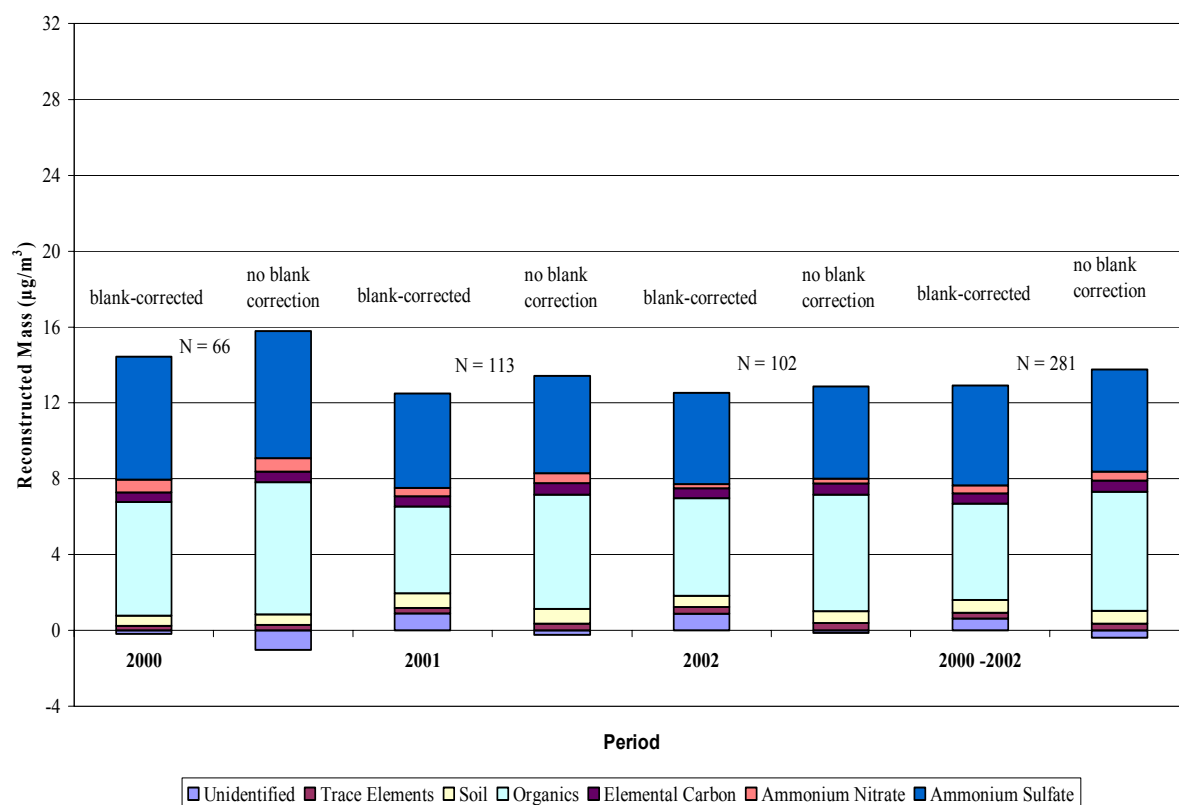
After blank subtraction, any negative values were changed to zero. Table 2 indicates the effect of the blank correction on the mean concentration of select species for the period 2000-2002. It shows the change for all STN and non-STN samplers and also shows which site had the greatest change for each species. For the major species obtained from the STN samplers, the blank correction resulted in a change of concentration that was usually less than 10%, but was as high as 58% for barium. For the trace species obtained from the STN sampler, the blank correction resulted in a change of concentration that varied widely, ranging from 1 to 65%, depending on species. For the major species obtained from the non-STN samplers, the blank correction resulted in a change of concentration that was usually less than 20%, but was as high as 52% for barium. For the trace species obtained from the non-STN samplers, the blank correction resulted in a change of concentration that varied widely, ranging from 1 to 65%, depending on species. The blank corrections for the trace species (all XRF elements) resulted comparable percent changes for both the STN and non-STN samplers. For organic carbon, the blank correction for STN samplers could represent up to 10% of the mean value while for the non-STN samplers it could represent up to 53%. For some of the minor species (in terms of mass) the blank values represented the entire uncorrected mean concentration (e.g., phosphorus at the Houston Aldine site). For species that are potentially toxic, failing to blank correct the concentration may result in overestimates of exposure and risk. Also, for species that are useful tracers for source apportionment, failing to blank correct when the blank values may vary by time, location, or sampler may lead to incorrect apportionment of these species in source/receptor modeling.

PM <sub>2.5</sub> Species	All STN Sites			STN Site w. Max. %Diff.			Site	All Non-STN Sites			Non-STN Site w. Max. %Diff.			Site
	BC Mean (µg/m <sup>3</sup> )	NBC Mean (µg/m <sup>3</sup> )	% Diff. (%)	BC Mean (µg/m <sup>3</sup> )	NBC Mean (µg/m <sup>3</sup> )	% Diff. (%)		BC Mean (µg/m <sup>3</sup> )	NBC Mean (µg/m <sup>3</sup> )	% Diff. (%)	BC Mean (µg/m <sup>3</sup> )	NBC Mean (µg/m <sup>3</sup> )	% Diff. (%)	
Major Species														
Mass	10.7160	11.0944	3.4	12.4871	13.0085	4.0	DALHIN	11.2251	11.6616	3.7	3.8094	4.2185	9.7	MCDOBS
Sulfate	2.8261	2.8579	1.1	1.3811	1.4079	1.9	ELPACH	3.4936	3.5720	2.2	3.3455	3.4854	4.0	CONROE
Non-volatile Nitrate	0.3972	0.4326	8.2	0.2459	0.2790	11.9	HSTDPC	0.2831	0.3217	12.0	0.0288	0.0543	47.0	MCDOBS
Ammonium	1.0631	1.0631	0.0	1.3626	1.3626	0.0	DALHIN	1.0122	1.0172	0.5	0.8972	0.9138	1.8	CONROE
Sodium Ion	0.1291	0.1372	5.9	0.0547	0.0620	11.8	ELPACH	0.1339	0.1425	6.0	0.0539	0.0688	21.6	ALPINE
Potassium Ion	0.0393	0.0414	5.0	0.0364	0.0390	6.7	HSTDPC	0.0504	0.0586	14.1	0.0488	0.0707	31.0	HSTHRM
Organic Carbon	2.6657	2.8979	8.0	2.2017	2.4527	10.2	HSTDPC	2.8302	3.5443	20.2	0.7942	1.6987	53.2	BIGBND
Elemental Carbon	0.4830	0.5132	5.9	0.3043	0.3353	9.2	HSTDPC	0.3813	0.4370	12.7	0.0443	0.1098	59.6	MCDOBS
Aluminum	0.0867	0.0874	0.9	0.0547	0.0561	2.4	DALHIN	0.0705	0.0714	1.2	0.0257	0.0274	6.3	MCDOBS
Barium	0.0067	0.0160	58.3	0.0058	0.0207	71.9	HSTDPC	0.0083	0.0173	52.0	0.0008	0.0056	85.5	MCDOBS
Calcium	0.1101	0.1112	1.0	0.0536	0.0551	2.8	HSTDPC	0.0757	0.0777	2.6	0.0470	0.0499	5.8	MRCVIL
Chlorine	0.0282	0.0283	0.4	0.0038	0.0040	3.9	DALHIN	0.0323	0.0326	0.8	0.0006	0.0006	9.1	ALPINE
Iron	0.0846	0.0861	1.8	0.0574	0.0591	2.9	HSTDPC	0.0816	0.0840	2.8	0.0156	0.0183	14.5	MCDOBS
Magnesium	0.0178	0.0186	4.7	0.0061	0.0074	17.5	HSTDPC	0.0149	0.0154	3.6	0.0034	0.0039	14.3	HSTHRM
Potassium	0.0699	0.0701	0.4	0.0616	0.0620	0.7	HSTDPC	0.0758	0.0764	0.7	0.0163	0.0169	3.5	MCDOBS
Silicon	0.2397	0.2432	1.4	0.1822	0.1859	2.0	HSTDPC	0.2069	0.2132	2.9	0.1103	0.1187	7.1	BIGBND
Sodium	0.0957	0.1056	9.4	0.0406	0.0476	14.7	ELPACH	0.1021	0.1115	8.4	0.0882	0.1032	14.5	HSTHRM
Sulfur	0.9452	0.9490	0.4	0.4785	0.4827	0.9	ELPACH	1.1276	1.1349	0.6	0.9514	0.9677	1.7	DONAPK
Trace Species														
Arsenic	0.0008	0.0008	9.9	0.0005	0.0006	14.2	DALHIN	0.0012	0.0012	7.3	0.0002	0.0002	24.3	BIGBND
Cadmium	0.0006	0.0009	39.2	0.0004	0.0010	57.3	ELPACH	0.0006	0.0010	36.2	0.0005	0.0011	54.6	MCDOBS
Copper	0.0034	0.0043	21.4	0.0019	0.0030	35.9	HSTDPC	0.0026	0.0037	30.1	0.0004	0.0029	84.9	MCDOBS
Lead	0.0029	0.0033	12.5	0.0016	0.0024	32.2	HSTDPC	0.0025	0.0029	14.0	0.0007	0.0011	36.8	ALPINE
Manganese	0.0019	0.0021	8.8	0.0016	0.0018	11.2	DALHIN	0.0021	0.0023	8.5	0.0008	0.0010	20.1	BIGBND
Mercury	0.0003	0.0004	33.0	0.0002	0.0004	41.1	HSTDPC	0.0004	0.0005	27.6	0.0002	0.0005	58.5	HSTDPA
Nickel	0.0012	0.0013	7.9	0.0002	0.0004	33.8	ELPACH	0.0011	0.0012	9.6	0.0002	0.0003	35.0	ALPINE
Phosphorus	0.0012	0.0018	33.1	0.0003	0.0005	38.5	HSTDPC	0.0005	0.0007	32.2	0.0000	0.0002	100.0	HSTALD
Selenium	0.0005	0.0006	15.7	0.0002	0.0003	41.2	ELPACH	0.0005	0.0006	15.0	0.0001	0.0002	40.1	BIGBND
Tin	0.0019	0.0054	64.5	0.0015	0.0063	76.9	HSTDPC	0.0019	0.0055	64.5	0.0011	0.0081	86.5	HSTDPA
Vanadium	0.0019	0.0020	5.7	0.0008	0.0009	13.8	DALHIN	0.0026	0.0027	4.6	0.0008	0.0010	19.8	ALPINE
Zinc	0.0090	0.0091	1.1	0.0089	0.0091	1.4	DALHIN	0.0096	0.0097	1.4	0.0008	0.0010	15.0	MCDOBS

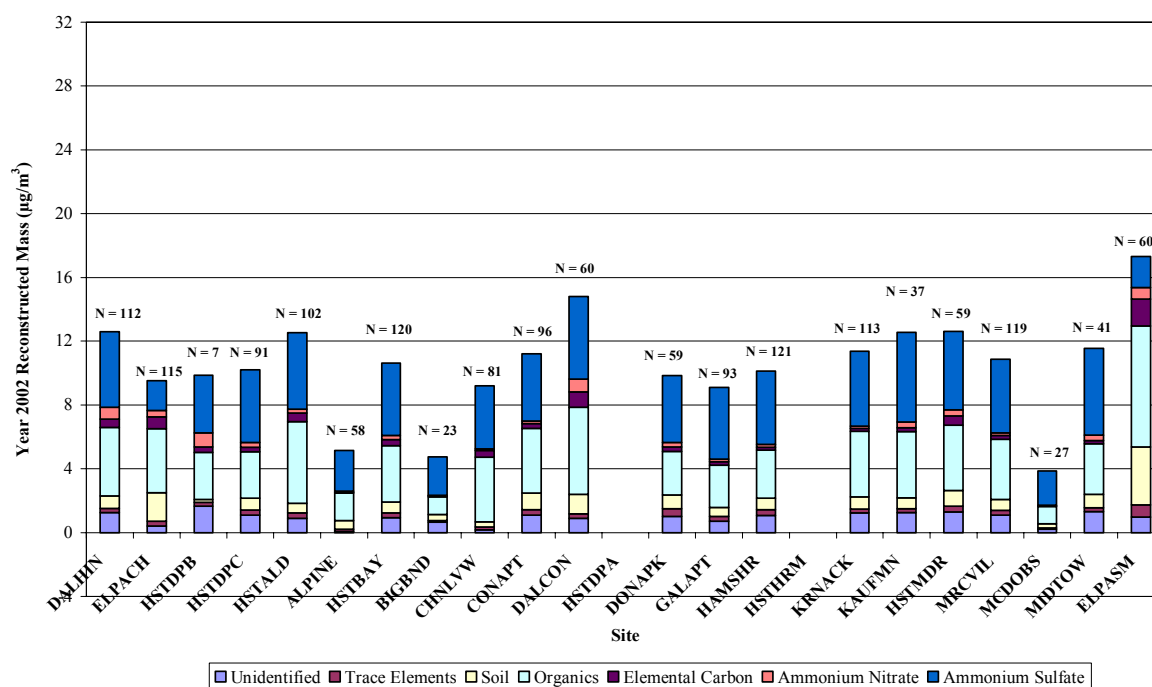
**Table 2. The effect of blank correction on summary statistics of select constituents for STN and non-STN speciation sites for the period 2000-2002.**

Using information on laboratory errors, MDLs, and sampler volumes provided by RTI, the estimated uncertainties were calculated for each species' concentration using a propagation of errors technique. [9] These calculated uncertainties compared favorably with the collocated precision results for Houston Deer Park. As one would expect, the uncertainties are dominated by the error in the sample volume and replicate precision when concentrations are well above the limits of quantification, but are dominated by the error associated with the MDLs when the concentrations are comparable to or less than the limits of quantification.

When reconstructed mass (RCM) was calculated with and without blank correction for the same dates, blank correction generally reduced the RCM for STN samplers by less than 5% annually and 10% quarterly; for the non-STN samplers the comparable reductions were about 10% annually and 20% quarterly. Figure 5 shows the effect for the non-STN sampler at the Houston Aldine site for the period 2000-2002. When the unidentified portion of the RCM for the data without blank correction is negative, the RCM accounted for more than 100% of the measured mass. Figure 6 gives an overview of the blank-corrected RCM for all sites for the year 2002.



**Figure 5. Effect of blank correction on the annual average reconstructed mass for the non-STN sampler at the Houston Aldine site.**



**Figure 6. Average blank-corrected, reconstructed mass for all sites for the year 2002.**



The West Texas sites have significantly lower annual averages for PM<sub>2.5</sub> mass than the other sites. Table 3 shows components of the RCM as a percentage of the measured mass in select areas of Texas for the same year and indicates that, with blank correction, the RCM accounts for about 92-95% of the measured mass.

	<i>El Paso</i>	<i>West Texas</i>	<i>Dallas</i>	<i>Houston</i>
Unidentified	5.2 ± 1.0	6.9 ± 6.3	8.1 ± 3.0	7.9 ± 3.7
Trace Elements	3.9 ± 1.0	2.3 ± 0.4	2.1 ± 0.1	2.9 ± 0.5
Soil	20.6 ± 2.0	8.6 ± 2.2	7.3 ± 1.5	6.0 ± 1.8
Organics	44.6 ± 1.6	28.8 ± 5.0	36.4 ± 2.0	36.8 ± 6.4
Elemental Carbon	9.1 ± 1.5	1.4 ± 0.2	5.3 ± 1.6	4.1 ± 0.7
Ammonium Nitrate	4.4 ± 0.1	1.1 ± 0.2	5.8 ± 0.2	2.3 ± 0.8
Ammonium Sulfate	16.1 ± 1.0	53.2 ± 3.4	37.1 ± 1.8	42.8 ± 2.9
Reconstructed Mass	94.8 ± 1.0	93.1 ± 6.3	91.9 ± 3.0	92.1 ± 3.7
<b>Mass (µg/m<sup>3</sup>)</b>	<b>12.9 ± 5.2</b>	<b>4.5 ± 0.6</b>	<b>13.4 ± 1.5</b>	<b>10.7 ± 1.4</b>

**Table 3. Components of reconstructed mass as a percentage of the measured PM<sub>2.5</sub> mass in select areas of Texas for 2002.**

## CONCLUSIONS AND RECOMMENDATIONS

The results of several quality and data assessments of PM<sub>2.5</sub> chemical speciation data at 23 sites in Texas during the three-year period 2000-2002 highlight issues that could affect regional or other large-scale monitoring projects. These include potentially adverse effects on: (1) the comparability of data due to the use of different sampling or monitoring instruments, analytical laboratories or instruments, and treatment of the data (e.g., if or how data were blank corrected and how values near or below detection limits are treated); (2) the accuracy of the data due to similar factors; and (3) the usefulness of data due to missing elements (e.g., MDLs and uncertainties for source/receptor models) and potential artifacts in trend analyses due to inadequate correction for changes in methodologies or blank levels over time.

Regional or large-scale monitoring projects should try to minimize these potential adverse impacts by adequate planning, including preliminary and ongoing quality assurance/quality control (QA/QC) assessments and discussions among data users. The effect of different sampling and analytical methodologies should be minimized or at least carefully assessed. The intended uses of the data should be derived from all the likely end-users and reflected in the elements included in the database and the processing of those elements before inclusion in the database. A pilot study with about 10-20% of the sites (reflecting as wide a range of ambient conditions as possible) should be conducted prior to the full network or study, the preliminary study data assessed by users and a QA team and any necessary corrections and/or adjustments made before full implementation. Part of the pilot study and ongoing QA/QC assessments should include inter-comparison sites with at least two monitoring instruments of each type used and laboratory inter-comparisons among all analytical laboratories, including evaluations of filter media and blank levels.

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