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## **INTRODUCTION**

In 1992, the California Air Resources Board's (CARB) Monitoring and Laboratory Division initiated particulate matter size selective inlet mass analysis system audits. The system audits' focus was on laboratories in California that conducted mass analysis measurements of particulate matter having an aerodynamic diameter less than or equal to a nominal 10 micrometers (PM10) in ambient air. The system audits incorporated performance audits of the balances used to weigh the PM10 filters as well. These audits joined the field performance flow audits for PM10 samplers that were initiated in 1985. The system audits have identified several common problems encountered by the laboratories, many of which resulted in the invalidation of several years of valuable data. Given the problems discovered, and the impending promulgation of new and even more exacting requirements for a fine particulate matter (PM2.5) National Ambient Air Quality Standards (NAAQS), laboratories need to be prepared to conduct consistent particulate matter mass analyses in the future.

## **REQUIREMENTS**

California regulation requires that air quality data must meet State and federal requirements in order to be considered as "data-for-record". The CARB is responsible for enforcement of this regulation which for PM10 weighings requires that they be conducted in accordance with approved quality control practices per the United States Environmental Protection Agency's (U.S. EPA) 40 CFR, Part 58<sup>1</sup>. In order to meet this regulation, laboratories conducting filter weighings were advised to comply with the requirements found in U.S. EPA's 40 CFR, Part 50, Appendix J<sup>2</sup> and the guidelines outlined in U.S. EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II<sup>3</sup>.

## **HISTORY**

In 1985, CARB's Quality Assurance Section (QAS) initiated annual field performance flow audits for PM10 samplers. The flow audits established warning limits of +/-7% to +/-10% and control limits of +/-10%. Additionally, the samplers' true flows had to meet the required cut point range of +/-10% from a designed actual flow rate of 40.0 cubic feet per minute. The PM10 flow audits results periodically led to the invalidation of PM10 data.

In 1992, CARB's QAS initiated a PM10 mass analysis system audit program. The program began with QAS contacting the laboratories conducting PM10 mass analysis to discuss U.S. EPA requirements and how the regulations related to the local protocol. QAS staff discovered that the weighing procedures varied widely among the laboratories. Several laboratories appeared to meet U.S. EPA requirements, while others could not.

At the start of the PM10 program most filters came to a central laboratory for mass analysis. As more PM10 samplers came on line, more laboratories initiated filter weighings. CARB's QAS informed the local laboratories that if they intended to submit data as data-for-record, they needed to comply with State and federal requirements. Therefore, QAS initiated system audits to ensure the local laboratories' data quality.

In August of 1992, QAS conducted its initial system audit on CARB's Inorganics Laboratory Section's PM10 mass analysis laboratory. The laboratory served as a central weighing facility for a number of local entities. The system audit revealed that the data generated by the laboratory met all the requirements outlined by U.S.EPA. However, some data were invalidated in the mid-1980s due to control limit exceedances during equilibration and missing quality control records. From this initial system audit, QAS refined the audit procedures and prepared to expand the audit program.

In March of 1993, all California air pollution control districts were provided copies of quality assurance/quality control guidelines for the mass analysis of PM10 filters. The policy summary addressed the program rationale and the relationship between State and federal requirements. The districts were also informed that QAS would initiate system audits on their laboratories later that year.

PM10 mass analysis system audits of district laboratories were initiated during the fourth quarter of 1993. To date, twelve PM10 mass analysis system audits have been conducted. The audits evaluated the quality of the data already submitted to CARB and U.S. EPA<sup>4</sup>. Only data that met State and federal requirements could be used in actions taken pursuant to the Federal Clean Air Act of 1990 and the California Clean Air Act.

## **SYSTEM AUDIT DESCRIPTION**

A system audit entails completion of a PM10 laboratory operations system audit questionnaire and an on-site inspection and assessment of staff, facilities, quality control programs, data, and document control. The system audit also includes a performance audit consisting of an on-site check and assessment of the PM10 filter weighing balance, relative humidity and temperature sensors, and documentation. Performance audits are conducted annually following the initial system audit.

A detailed system audit report is issued following the on-site inspection and assessment of the laboratory. The system audit report includes the following: executive summary; conclusion; recommendations; system audit objectives; organization; laboratory facility and operations; data management; quality assurance and quality control; performance audit; data quality; and follow-up.

## **FINDINGS**

Below is a list of the most common problems found while conducting PM10 mass analysis system audits. Note that not all of the laboratories had all of the deficiencies identified. Several

of the problems were the result of laboratory staff being unfamiliar with or not understanding the requirements, and/or not having the resources to satisfy the requirements.

1. Filter equilibration was not repeated prior to weighings when the relative humidity or temperature in the weighing/equilibration room exceeded the requirements. Also, the equilibration results were oftentimes not recorded.
2. Tare and gross weight checks (duplicate weighings) were not conducted on each day of filter weighing. This information could have been used to support the data when the relative humidity or temperature in the equilibration/weighing room exceeded the limits, thereby preventing the invalidation of PM10 data.
3. Filters found to be outside the range of 3.7 to 4.7 grams during initial weighing were not investigated and were still used to collect samples.
4. Filter weighing balances were not calibrated at least annually as required.
5. Standard weight checks of the balance were not conducted and the results were not recorded on each day of filter weighing.
6. Laboratory staff did not notify their immediate supervisor and appropriate corrective action was not taken and documented when standard weight checks exceeded requirements.
7. Standard operating procedures, calibration reports, log book/sheets, and U.S. EPA guidelines were not kept in the equilibration/weighing room, were not accessible to staff, were missing, or were incomplete.
8. The relative humidity and temperature sensors were not calibrated (checked) semiannually against a wet bulb/dry bulb psychrometer or equivalent and against a National Institute of Standards and Technology (NIST) certified thermometer, respectively. Also, the results of the checks were oftentimes not recorded in a calibration log book or on calibration log sheets.
9. A chain-of-custody document did not accompany each filter at all times. Often the document was missing or incomplete.
10. Log books and/or log sheets lacked pertinent information. Missing information included duplicate weighings, temperature, relative humidity, daily balance checks, dates, initials, etc.
11. Make-up runs were not scheduled when a PM10 sample failed or was invalidated to ensure that U.S. EPA and CARB data representativeness air quality measurement and statistics criteria were satisfied.

## **IMPLICATIONS**

Several of the laboratory mass analysis deficiencies, including poor record keeping; inadequate filter equilibration (relative humidity and temperature); and missing duplicate weighings and balance calibrations, resulted in the invalidation of several years of valuable data. Approximately 20% of the PM10 data, since 1985, from over 100 samplers were invalidated due to problems with mass analysis. Some of the 12 laboratories audited had no data invalidated, while others had more than 50% of their data invalidated. These percentages do not include PM10 data invalidated as a result of field audits of the samplers. The focus of the PM10 mass analysis audits was not to invalidate data, but rather to ensure the data generated and submitted to CARB and U.S. EPA were of the highest quality. It is to the advantage of each laboratory

conducting particulate matter mass analyses to observe U.S. EPA requirements and guidelines and to keep well documented records to ensure the data being generated are of good quality and can be considered data-for-record. This is especially critical for those areas seeking attainment designation. With U.S. EPA's impending promulgation of a PM<sub>2.5</sub> mass analysis program, laboratories need to be prepared to conduct consistent particulate matter mass analyses. Many of the proposed requirements for PM<sub>2.5</sub>, covering the areas of sampler operation and mass analysis, are more numerous and more strict than those required of PM<sub>10</sub>. Laboratories that undertake PM<sub>2.5</sub> weighings as a means of generating data-for-record need to thoroughly familiarize themselves with the essential (and discretionary) PM<sub>2.5</sub> mass analysis requirements once they are approved. The experience with our system audits tells us that laboratory failures are largely the result of a lack of understanding of the regulation. In some instances, small entities have been unable to fully fund environmental chambers or recording sensors. Given the greater emphasis U.S. EPA is placing on field and laboratory quality control, local districts would be advised to fully understand the cost and procedures that are entailed to obtain acceptable results.

## **CONCLUSIONS**

The objective of the PM<sub>10</sub> mass analysis system audits was to validate the quality of PM<sub>10</sub> data generated and submitted by laboratories to U.S. EPA and CARB. The audits identified several common problems which, if corrected, would improve the overall quality of laboratories' PM<sub>10</sub> mass analysis programs. Several of the problems, including tolerance exceedances and inadequate record keeping of performance parameters (that could have in fact been good), resulted in the invalidation of nearly 20% of valuable PM<sub>10</sub> data since 1985. With the impending promulgation of a PM<sub>2.5</sub> program, laboratories need to be better prepared to meet the challenge of performing consistent particulate matter mass analyses in the future. Avoiding the problems identified with PM<sub>10</sub> mass analysis during the early stages of the PM<sub>2.5</sub> program is critical and will prevent valuable data from being invalidated. Therefore, it is imperative that laboratories ensure their programs are in compliance prior to implementation by adhering to the requirements and guidelines.

## **REFERENCES**

1. 40 CFR, Part 58; Ambient Air Quality Surveillance; U.S. Environmental Protection Agency, July 1991.
2. 40 CFR, Part 50, Appendix J; Reference Method for the Determination of Particulate Matter as PM<sub>10</sub> in the Atmosphere; U.S. Environmental Protection Agency, July 1991.
3. Reference Method for the Determination of Particulate Matter as PM<sub>10</sub> in the Atmosphere (High Volume PM<sub>10</sub> Sampler Method); U.S. Environmental Protection Agency, Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Section 2.11.4, January 1990.
4. Loscutoff, W.V.; Quality Assurance/Quality Control Guidelines for the Mass Analysis of PM<sub>10</sub> Samples; California Air Resources Board, March 26, 1993.