

9.0 Analytical Methods

The choice of methods used for any EDO should be influenced by the DQO. From the DQO and an understanding of the potential population uncertainty, one can then determine what measurement uncertainty is tolerable and select the method most appropriate in meeting that tolerance. Methods are usually selected based upon their performance characteristics (precision, bias, limits of detection), ease of use, and their reliability in field and laboratory conditions.

Since both field and analytical procedures have been developed for the criteria pollutants in the Ambient Air Quality Monitoring Program, and in the various technical assistance documents for the other national ambient air programs, this section will discuss the general concepts of standard operating procedures and good laboratory practices as they relate to the reference and equivalent methods. A more detailed discussion on the attributes of SOPs can be found in Section 5. Information on reference and equivalent methods can be found on the AMTIC website¹ as well as the current list of designated Federal Reference and Equivalent Methods².

Many ambient air methods utilize continuous instruments and therefore do not involve laboratory analysis. However particulate matter methods involve both continuous and manual methods and some of the other major monitoring programs involve sampling which requires the use of laboratory analysis. Table 9-1 provides a summary of the pollutants measured and the analytical methods for these programs.

Table 9-1 Acceptable Analytical Methods

| Network | Pollutant | Acceptable Method | Reference |
|---------|---|--|----------------------|
| SLAMS | PM ₁₀ – Hi-Vol | Gravimetric | 40 CFR Part 50 App B |
| SLAMS | PM ₁₀ - dichot | Gravimetric | 40 CFR Part 50 App J |
| SLAMS | PM _{2.5} | Gravimetric | 40 CFR Part 50 App L |
| SLAMS | PM _{10-2.5} | Gravimetric- difference | |
| SLAMS | Pb | Atomic Absorption Spectrometry | 40 CFR Part 50 App G |
| PAMS | VOCs | Gas Chromatography/Mass Spectrometry (GC/MS) | TO-15 |
| PAMS | Carbonyl compounds | High Performance Liquid Chromatography (HPLC) | TO11-A |
| PAMS | Non-Methane Organic Compounds (NMOC) | Cryogenic Preconcentration and Direct Flame Ionization Detection (PDFID) | TO-12 |
| NATTS | Metals | Inductively coupled plasma (ICP) | IO 3.5 |
| NATTS | Aldehydes | High Pressure Liquid Chromatography | TO11-A |
| NATTS | VOCs | Gas Chromatography | TO-15 |
| STN | PM _{2.5} | Gravimetric | 40 CFR Part 50 App L |
| STN | Elements | Energy Dispersive X-Ray Fluorescence (EDXRF) | STN QAPP and SOPs |
| STN | Anions | | STN QAPP and SOPs |
| STN | Cations | | STN QAPP and SOPs |
| STN | Organic, Elemental, Carbonate, Total Carbon | Thermal Optical Carbon Analyzer | STN QAPP and SOPs |
| STN | Semi-volatile Organic Compounds | Gas Chromatography/Mass Spectrometry (GC/MS) | STN QAPP and SOPs |

The SLAMS network provides more rigorous quality control requirements for the analytical methods. These methods are found in 40 CFR Part 50, as described in the references. In addition, the method identified for Pb is the reference method. There are a number of equivalent analytical methods that are

¹ <http://www.epa.gov/ttnamti1/pmfrm.html>

² <http://www.epa.gov/ttn/amtic/criteria.html>

available for the Pb. Some of the NATTS methods are derived from the Toxics Organic Method Compendium³. Others, like the STN Network⁴ may be developed specifically for the program, based on the national laboratory currently performing the analysis. The PAMS, NATTS and STN networks follow the performance based measurement process paradigm. These Networks' QA project plans or technical assistance documents suggest a method, but also allow some flexibility to use other methods that meet the network's measurement quality objectives. Various, independent proficiency test samples and technical systems audits are performed to ensure that the data quality within these networks remains acceptable.

9.1 Good Laboratory Practices

Good laboratory practices (GLPs)⁵ refer to general practices that relate to many, if not all, of the measurements made in a laboratory. They are usually independent of the SOP and cover subjects such as maintenance of facilities, records, sample management and handling, reagent control, and cleaning of laboratory glassware. In many cases, the activities mentioned above may not be formally documented because they are considered common knowledge. However, for consistency in laboratory technique, these activities should have some form of documentation.

9.2 Laboratory Activities

For ambient air samples to provide useful information or evidence, laboratory analyses must meet the following four basic requirements:

1. Equipment must be frequently and properly calibrated and maintained (Section 12).
2. Personnel must be qualified to make the analysis (Section 4).
3. Analytical procedures must be in accordance with accepted practice (Section 9.1 above).
4. Complete and accurate records must be kept (Section 5).

As indicated, these subjects are discussed in other sections of this document. For the Ambient Air Quality Monitoring Program, laboratory activities are mainly focused on the pollutants associated with manual measurements for lead, particulate matter (PM and STN), NATTS⁶ and PAMS⁷ (VOCs). However, many laboratories also prepare reference material, test or certify instruments, and perform other activities necessary to collect and report measurement data. Each laboratory should define these critical activities and ensure there are consistent methods for their implementation.

³ <http://www.epa.gov/ttn/amtic/airtox.html>

⁴ <http://www.epa.gov/ttn/amtic/specsop.html>

⁵ <http://www.epa.gov/Compliance/monitoring/programs/fifra/ghp.html>

⁶ http://www.epa.gov/ttn/amtic/files/ambient/airtox/NATTS_TAD_SECT_4.pdf

⁷ <http://www.epa.gov/ttn/amtic/files/ambient/pams/newtad.pdf>