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Technical Note- Holding Time Requirements for PM2.5 Filter Samples

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There has been some confusion on the proper interpretation of the holding time window for PM2.5 filter samples that have been collected within the 177-hour retrieval time and whether one has 10 or 30 days to weigh these filter samples when the filter samples arrive at laboratories¹ either over the 25°C limit or over the average sample temperature during collection. The following provide additional guidance and interpretation to the rules in 40 CFR Part 50 Appendix L Section 10.13 and Section 8.3.6.

NOTE- This technical note supersedes the January 19, 2000 memorandum from David Mobley titled: "Additional Guidance on PM2.5 Cassette Handling and Transportation"

Before getting to the interpretation, there are a few recommendations that can help avoid the situations that put filter samples at risk of being invalidated.

Use of Ice Substitutes, Documentation of Sample Container Temperatures and Post-Sampling Conditioning and Weighing.

Ice substitutes- Use of ice substitutes are recommended for direct transport as well as overnight transport. For direct, same day transport to laboratories, use of ice substitutes will ensure that filter sample cooling has started and does not add to transportation costs. For overnight transport, ice substitutes ensure that the process of cooling the filter sample has started and if shipments are delayed, temperatures in the sample containers, once documented at the laboratory, more than likely will meet acceptance criteria or minimally be acceptable for a 10-day weigh regime without calling the validity of the filter sample(s) into question.

Documentation of Sample Container Temperature- Max/min thermometers are recommended for use. These types of thermometers provide the appropriate documentation of sample container temperature during transport. Recording and documenting a sample container temperature upon arrival at the laboratory provides a reasonable measurement of sample container temperature, especially when ice substitutes are used. However, if a filter sample shipment is delayed for any number of days, and ice substitutes are not used (or have completely thawed during the delayed shipment), one cannot determine if temperature fluctuation above acceptance limits occurred within the shipping container. A temperature measurement upon arrival at the laboratory may only indicate current temperature of the container, whereas a max/min thermometer allows you to discern if the filters stayed within the appropriate temperature range throughout transport.

Post-sampling Conditioning and Weighing – There appear to be cases where filter samples arrive at the lab and are placed directly into the conditioning environment, even if they are not being weighed within 24-48 hours (or however long it minimally takes for the filter samples to equilibrate in a particular laboratory). It

¹ A laboratory, in the context of this document, is a facility that includes sample receipt, cold storage, sample conditioning and the gravimetric weighing laboratory.

has not been the intent for filter samples to be placed in a conditioning environment any longer than necessary to equilibrate since they may be subject to additional contamination or temperatures not measured during sampling. If filter samples arrive at the laboratory in a manner that they can be post-weighed 10 or 30 days from the end of the sample period, they are expected to be placed in cold storage² until the time when they are being prepared for weighing. Only at this point should they be removed from cold storage and placed in the conditioning environment.

Interpretation of the Appendix L Requirements

The parts of the regulation concerning filter collection, conditioning, and weighing that have caused some confusion are 40 CFR Part 50 Appendix L Section 10.13 and Section 8.3.6.

10.13 *After retrieval from the sampler, the exposed filter containing the PM_{2.5} sample should be transported to the filter conditioning environment as soon as possible, ideally to arrive at the conditioning environment within 24 hours for conditioning and subsequent weighing. During the period between filter retrieval from the sampler and the start of the conditioning, the filter shall be maintained as cool as practical and continuously protected from exposure to temperatures over 25 °C to protect the integrity of the sample and minimize loss of volatile components during transport and storage. See section 8.3.6 of this appendix regarding time limits for completing the post-sampling weighing.*

8.3.6 *The post-sampling conditioning and weighing shall be completed within 240 hours (10 days) after the end of the sample period, unless the filter sample is maintained at temperatures below the average ambient temperature during sampling (or 4 °C or below for average sampling temperatures less than 4 °C) during the time between retrieval from the sampler and the start of the conditioning, in which case the period shall not exceed 30 days. Reference 2 in section 13.0 of this appendix has additional guidance on transport of cooled filters.*

These two sections contain important temperature requirements that determine if a filter sample can be post-weighed 30 days after the end of the **sample period** or 10 days after the end of the **sample period**. A sample period is the 24-hour midnight to midnight sampling period for a filter. Therefore, the end of a sample period is typically midnight and the post-sampling conditioning and weighing period starts at midnight (the end of the sample period). The term “post-sampling conditioning and weighing” is the period of time, either 10 or 30 days, that a filter sample must be weighed after sampling of that filter sample has ended (sampling period). Further down in section 8.3.6 it is simply referred to as “period”. Usually a filter sample is retrieved from a sampler, shipped/transported to a lab, set in cold storage, removed from cold storage, conditioned for approximately 24-48 hours, and then weighed.

The following edits to 8.3.6 do not change the intent of the regulation but provides more clarity:

*The post-sampling conditioning and weighing **period** shall be completed within 240 hours (10 days) after the end of the sample period, unless the filter sample is maintained at temperatures below the average ambient temperature during sampling (or 4 °C or below for average sampling temperatures less than 4 °C) during the time between retrieval from the sampler and the start of the conditioning, in which case the **post-sampling conditioning and weighing period** shall not exceed 30 days. Reference 2 in section 13.0 of this appendix has additional guidance on transport of cooled filters.*

The following three scenarios are used to assist monitoring organizations in interpreting when the post-sampling conditioning and weighing period can be 30 days and when it must be 10 days. In these scenarios, it assumes that, minimally, the temperature of the sample container is measured upon arrival at

² Cold storage in this document will be a refrigerator set between 0°C and 4°C, not frozen

the laboratory or recorded on a max/min thermometer (preferred) that is placed within the sample container during transport. Figure 1 at the end of this discussion captures the decisions made in these three scenarios.

Scenario #1 Average Sample Temperature Greater than 25°C

In this scenario the average sample temperature for a 24-hour sample period for a particular filter sample is 30°C. In this case, the filter sample can be given a 30-day post-conditioning and weighing period holding time from the end of the sample period if:

1. The filter sample is transported directly (same day) to the lab in a sample container and it can be documented³ that the sample container did not exceed the average sample temperature (i.e., 30°C). The filter sample must then be immediately placed in cold storage until conditioning.
2. The filter sample is placed in a sample container with an ice substitute for overnight transport and the documented temperature of the sample container upon arrival at the laboratory⁴ is equal to or less than 25°C. The filter sample must then be immediately placed in cold storage until conditioning.
3. The filter sample was placed in a sample container with a max/min thermometer and the max temperature of the sample container upon arrival at the lab is equal to or less than 25°C. The filter sample must then be immediately placed in cold storage until conditioning.

If, however:

- no ice substitutes were used or were used but the ice substitutes were at ambient temperature upon arrival to the lab,
- a max/min thermometer was used and the temperature in the sample container was greater than the average sample temperature or,
- no max/min thermometer was used,

then the filter sample in the container is now of questionable integrity since it cannot be documented or verified that the temperature in the sample container was maintained within acceptance limits during transport. Minimally, the filter sample needs to be weighed within 10 days from the end of its sampling period. The validity of the filter sample is in question.

Scenario #2 Average Sample Temperature Less Than 25°C and Greater than 4°C

In this scenario, the filter sample can be given a 30-day post-conditioning and weighing period holding time from the end of the sample period if:

1. The filter sample is transported directly (same day) to the lab in a sample container and it can be documented that the sample container temperature did not exceed the average sample temperature. The filter sample must then be immediately placed in cold storage until conditioning.
2. The filter sample is placed in a sample container with an ice substitute for overnight transport and the documented temperature of the sample container upon arrival at the laboratory did not exceed the average sample temperature and the ice substitute is still cold to the touch. The filter sample must then be immediately placed in cold storage until conditioning.
3. The filter sample is placed in a sample container with a max/min thermometer and the documented max temperature of the sample container upon arrival at the laboratory did not

³ Documented means that the sample temperature of the sample container is measured upon arrival at the laboratory and documented on chain of custody.

⁴ In cases where multiple samples are placed in one container without any ice substitute each sample must be evaluated separately based on the individual sample's average sample temperature.

exceed average sample temperature. The filter sample must then be immediately placed in cold storage until conditioning.

If the temperature in the sample container is above the average sample temperature, but below 25°C, the filter sample is acceptable but must be weighed within 10 days from the end of the sample period.

If, however:

- no ice substitutes were used, or were used but at ambient temperature upon arrival to the lab,
- a max/min thermometer was used but the temperature was greater than 25°C or,
- no max/min thermometer was used,

then the filter sample in the container is now of questionable integrity since it cannot be documented or verified that the temperature in the sample container was maintained within acceptance limits during transport. Minimally, the filter sample needs to be weighed within 10 days from the end of its sample period. The validity of the filter sample is in question

Scenario #3 Average Sample Temperature Less Than 4°C

In this scenario the average sample temperature is less than or equal to 4°C. The filter sample can be given a 30-day post-conditioning and weighing period holding time from the end of the sample period if:

1. The filter sample is transported directly (same day) to the lab in a sample container and it can be documented that the sample container upon arrival in the laboratory did not exceed the 4°C. The filter sample must then be immediately placed in cold storage until conditioning.
2. The filter sample was placed in a sample container with an ice substitute for overnight transport and the documented temperature of container upon arrival at the lab is equal to or less than the average sample temperature and the ice substitute is cold. The filter sample must then be immediately placed in cold storage until conditioning.
3. The filter sample was placed in a sample container with a max/min thermometer and the max temperature did not exceed 4°C upon arrival at the laboratory. The filter sample must then be immediately placed in cold storage until conditioning.

If the temperature in the sample container is above 4°C but below 25°C then the filter sample is acceptable but must be weighed within 10 days from the end of the sample period.

If, however:

- no ice substitutes were used, or were used but at ambient temperature upon arrival to the lab,
- a max/min thermometer was used but the temperature was greater than 4°C or,
- no Max/min thermometer was used,

then the filter sample in the container is of questionable integrity since it cannot be documented or verified that the temperature in the sample container was maintained within acceptance limits during transport. Minimally, the filter sample needs to be weighed within 10 days from the end of its sample period. The validity of the filter sample is in question

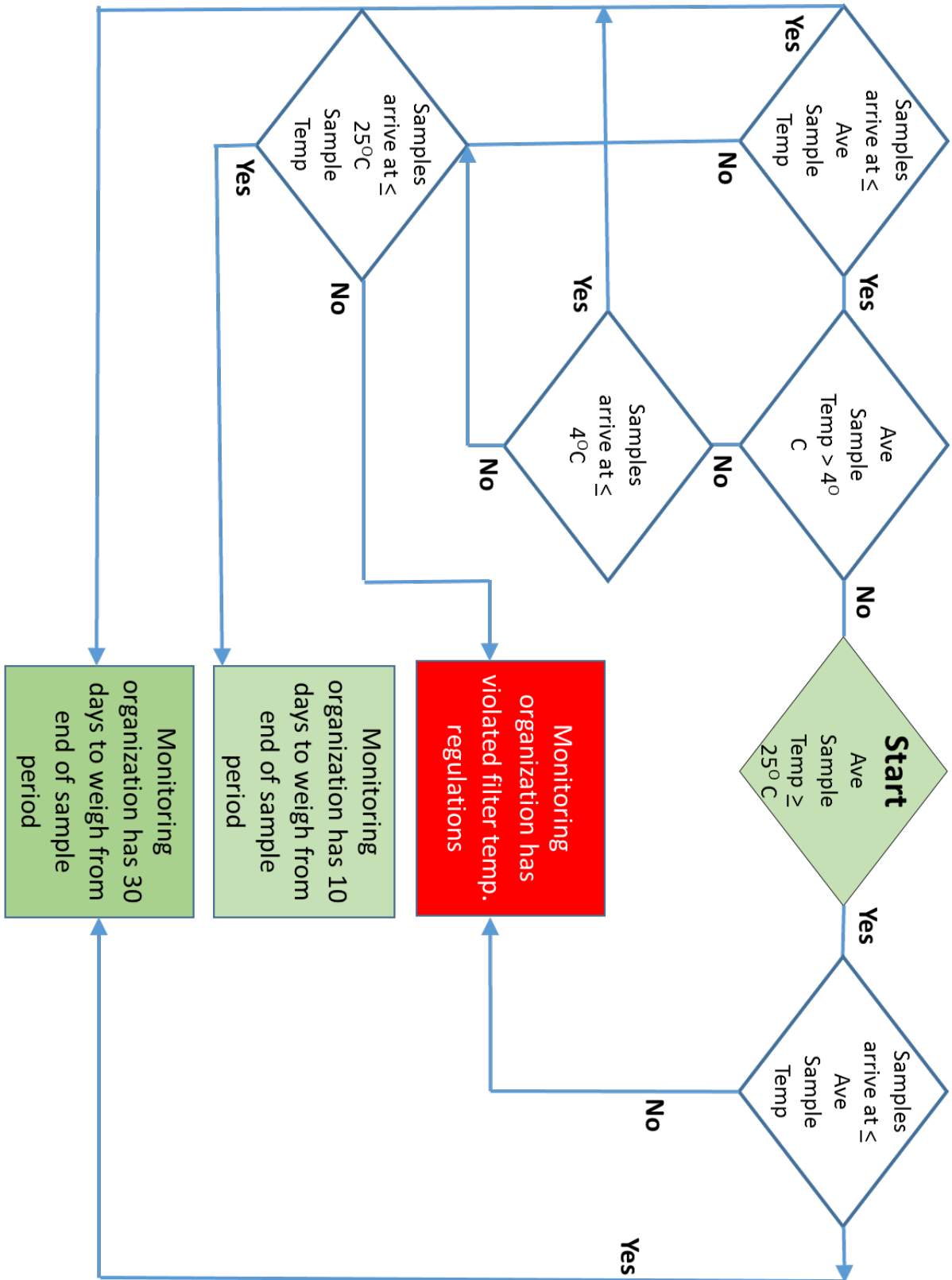


Figure 1. Filter Sample Holding Time Decision Flowchart