Response to Outside Review of NMAM Method 9106 (Methamphetamine and Illicit Drugs, Precursors, and Adulterants on Wipes by Liquid-Liquid Extraction)

Date: June 1, 2011
From: NIOSH Manual of Analytical Methods (NMAM) editors
Research chemist, Kevin Ashley
Chemist, Paula Fey O'Connor

Note: Editors' responses to reviewers are in red.

Reviewer: **INTERNAL REVIEWER**

Comments – Single blind independent laboratory evaluation
reviewed the single blind independent laboratory evaluation of NIOSH 9106 conducted by a NIOSH contract laboratory (Bureau Veritas). The evaluation used wipe samples spiked with methamphetamine and pseudoephedrine that were subsequently analyzed for methamphetamine, pseudoephedrine, amphetamine, ephedrine, norephedrine, and MDMA ($CH_2O_2C_6H_3CH_2CH(CH_3)NHCH_3$). Only significant quantities of methamphetamine and pseudoephedrine were found except for some minimal amounts of ephedrine (between the Limit of Detection (LOD) and the Limit of Quantitation (LOQ)) found on some wipes.
determined that LODs, LOQs, and recoveries of methamphetamine and pseudoephedrine to be acceptable for publication as a NIOSH method.

Response: No significant changes were made to the method or backup data report as a result of comments. The review comments will be included as additional background information as a link to the method since the Review comments are a concise summary of the single blind independent laboratory evaluation of the method required for publication in the NIOSH Manual of Analytical Methods (NMAM).

External Reviewer:

EXTERNAL REVIEWER #1

Comments

1. In general, I thought that everything was very well written and presented. The background portion provided by DataChem was also good and easy to understand, even for a non-chemist. I only have a few things to add to the method and a few comments. None of my comments would warrant major (or even minor for that matter) changes to the documents. Since the IH portion is the same (I believe) for all of the methods, I am including them on this one sheet.

Response: No changes needed.

2. The biggest comment that I have is the applicability of the method on porous items. I believe that you do provide the information that the efficacy of the methods depends upon what material is being sampled but the inability of wipe samples to adequately determine the concentration of meth on a porous surface is very poor. I have enclosed a report on recovery that we did for Utah where we found that the recovery rate from very porous surfaces is less than 20%. It also seems to depend upon how the surface is contaminated. If it is contaminated by an aerosol formed by evaporating the meth, then penetration into surfaces like painted dry wall is common. Even with methanol, only about 40% of the meth present in the drywall is released. In my opinion, using a wipe to measure meth in a carpet, popcorn ceiling, unpainted wood, clothes, etc. is a poor choice of sampling methods. Sending actual pieces of the material to the lab would be much better.

Response: The method was developed for sampling with surface wipes on appropriate surfaces. We can add that, for surfaces that do not lend themselves easily to surface wiping, pieces of these surfaces might have to be analyzed to obtain a more accurate measure of the methamphetamine or whichever drug is being measured. Also, other surface sampling methods, such as vacuum sampling[1], may be appropriate. Report that is mentioned will be added as a reference in the method.

3. I believe that the limit of detection is very good. We also conducted some testing to determine the ability of a laboratory to accurately determine the amount of meth in a cotton wipe (as well as some other media) and I have attached that report. In general, especially DataChem, provided results that were very close to the spike. The number of false positives or false negatives were very low, especially at DataChem.

Response: This reference will be added to the method or backup data report.

4. Regarding the background document, the material suggests that there is not a health-based standard and that is relatively correct, although California has developed a risk-based standard. Based on that standard, some states are revising their limits upward. You might want to check table 1 in the background report since I believe that Utah and maybe a few other states have recently changed the accepted standard. The accepted levels do seem to be in flux at this time.

Response: The Table in the method and backup report that lists the standards will be checked so that the limits stated will be current as of the publication date.

5. The Method requires that the sample be refrigerated during collection and shipping. The method does say, however, that the sample may be OK without refrigeration. Our experience has been that refrigeration was not necessary. The accuracy of the spike samples that we sent were as good un-refrigerated as they were refrigerated but we did ship all of the samples via overnight mail.

Response: The method will show that refrigeration is preferred but samples are stable if kept unrefrigerated.

6. The use of blotting surfaces during sampling on surfaces where the cotton gauze will catch may be a problem since if you have to blot; the return will be very poor based on our experience. Surfaces that are rough will not give up the meth easily.

Response: This method has been through a "partial" evaluation, which means that recoveries from an exposure setting have not been through a statistical evaluation along with independent testing. Focus has been on analysis of sampling materials themselves.

7. Regarding the use of methanol and isopropanol. We also found that either solvent worked well although we normally use methanol. There was a difference between sampling meth that had been dropped on the surface with a pipette and that which was aerosolized onto the surface. Aerosolized meth was not removed with distilled water as easily as was meth dropped onto the surface. This is likely due to penetration into drywall, etc.

Response: The partial evaluation that was done for this method did not include any generated atmospheres of methamphetamines along with other illicit drugs. The evaluation that was done entailed spike samples.

External Reviewer: **EXTERNAL REVIEWER #3**

Comments

1. There is a table in both the Backup Data Report (pg.7, Table I) and method (Table II) that provides maximum surface contamination limits. The units in both are expressed as "µ/area". The correct units to express are "µg/area."

Response: Corrections were made as indicated.

2. The method describes the use of a tumbler for performing both the initial extraction and subsequent liquid/liquid extraction. Are any other mechanical devices acceptable for use? Can mechanical agitation (shaker) be used provided it is mixing the sample well? I suggest comments be added regarding whether the method must be performed with a tumbler and if other equipment is an acceptable alternative, indicate this. This comment also applies to the subsequent two methods. This method is lacking in contrasting the procedure to subsequently developed methods 9109 and 9111. I like the discussion in Backup Data Report 9109 that has a table contrasting the three procedures as well as the in-depth discussion of the advantages one procedure has over the others. I suggest this be listed in all three procedures to help guide the analyst to select the procedure that will best suit their needs and available equipment.

Response: The discussion of the 3 methods from the NMAM Method 9109 back up data report will be included the NMAM 9106 method discussion. Other agitation methods may be appropriate; it is up to the user, however, to test alternative techniques and determine their

suitability. The agitation methods employed here in method development and evaluation can be viewed as representative.

Reviewer:

EXTERNAL REVIEWER #4

1. Solutions 1(b):

   Target analyte spiking solutions are prepared by diluting the stock solutions to about 200 µg/mL each in methanol.

   Comment: Although we traditionally have used MeOH, we are beginning to see some anecdotal evidence that the use of MeOH is resulting in slight negative bias in recoveries. Have other investigators seen similar results, and/or has the use of MeOH versus other polar solvents been investigated?

Response 1: The user check for this method did have a slight negative bias. The amount of the bias was considered to be within the acceptable limits for methods developed for the NMAM. The method has met the NIOSH evaluation criteria.

2. SAMPLING

   3. Prepare a rigid template from disposable cardstock or a sheet of Teflon® having either a 10 cm x 10 cm or 1 ft x 1 ft square hole cut. The template must be able to retain its shape during wiping to ensure that the areas wiped were either 100 cm2 or 1 ft2. Single-use disposable cardstock should be used because it eliminates the possibility for cross-contamination and the necessity to take a blank wipe between samples in step 5.

   As marginally addressed in number ten in the Appendix, the practice of specifying rigid templates is greatly restricting the selection of more appropriate surface locations. The use of rigid templates has resulted in a misconception that the templates are necessary for some unspecified reason. The net result is that specifying templates has resulted in the interference of sampling in a manner that would more appropriately meet specified data quality objectives.
   For example, in processing a crime scene, the investigator wants to sample a base of a metallic reading lamp with a smooth convoluted circular surface. The investigator knows that by sampling the lamp base, their specific data quality objective would be better served; however, the investigator (usually someone with no specific training in sampling) rejects the surface since the rigid template does not neatly fit over the desired surface. The investigator believes the use of the template is more important than selection of an appropriate surface and now prioritizes potential sampling locations, not on the basis of how well the surface meets the DQOs but rather, how well a rigid template would cover the surface.
   Finally, the use of rigid templates as a "magic" practice, is limiting law enforcement's ability to obtain better information by surfacing larger areas. The CSI personnel are not aware of the fact that there is nothing magical about 100 cm2 or one square foot, and any area, regardless of size may be sampled provided that the area is known.

Recommendation:

Recommend that the language be substituted with language that instructs the investigator to identify and appropriate surface location, then, delineate the surface with a known measurement, and sample the surface, recording the dimensions of the surface thus sampled. Indeed, it is entirely possible (and indeed sometimes necessary) to wipe first, and then determine the dimensions of the surface after the wipe has been collected.

Response 2: Wording will be modified to allow for wiping using acceptable techniques but emphasizing that the surface area is the important measure. A tape-defined sampling procedure has been described in ASTM D6966,[1] and language from that standard will be incorporated to allow for an alternative to template-assisted sampling.

3. SAMPLING:

5. Use gauze in sterile packaging to minimize the chance for cross-contamination which might more easily occur with open bulk packaged cotton gauze. The gauze wipes needed for the laboratory media blanks and QC samples are to be sent to the laboratory in their unopened sterile packages.

The language creates a QA/QC problem in that the specificity requires the investigator to essentially identify the QA/QC sample to the laboratory. As an analyst, I know that if my blanks didn't blank out, I would adjust the run until they, and other QA/QC checks are within tolerances; this is a common, subconscious practice. The integrity and the probative value of the field blank is greater when the blank is handled in exactly the same manner as all other samples and is serendipitously submitted to the laboratory with no identifier to alert the laboratory to the identity of the sample. In our practice, a suite of sample wipes is prepared off-site. The samples are labeled and the identity of the field blank is unknown until the sampling begins – at which time, a specified number of containers are randomly withdrawn from the set for inclusion as blanks. Where surfaces areas are submitted, a fictitious surface area is also submitted for the blank so the laboratory is not alerted to the identity of the blank.

Finally, there is no information to support the statement: "Use gauze in sterile packaging to minimize the chance for cross-contamination which might more easily occur with open bulk packaged cotton gauze" I currently have well over 75 consecutive field blanks, all prepared from rolled gauze; prepared off-site, and I have never seen a contamination issue. This language should be removed.

Recommendation:

The language should be altered such that the field blank is prepared in an exact manner as the other samples, and is submitted in the sample suite without identifying the QA/QC sample.

Response 3: The reviewer does not seem to understand that in a typical lab, clean media are used to make up samples for blank corrections and recovery studies.
The field blank is a different sample that the industrial hygienist does not need to identify to the lab, but lets the IH know if contamination was introduced accidentally to the samples at some point between sampling and shipping to the analytical lab.
As for the "use of sterile gauze" for sampling, the wording will be modified to suggest that using sterile gauze is one way of assuring that no cross contamination will be introduced to the samples. For NIOSH Methods, QA/QC samples are samples that the lab's QA director adds for

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[1] ASTM D6966, *Standard Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Determination of Metals.* ASTM International: West Conshohocken, PA, 2003.

an internal lab check; such samples are typical in carrying out any standardized analytical methodology.

4. SAMPLING:

6. Secure the template(s) to the area(s) to be wiped (e.g. with tape along outside edge of template). If a single-use disposable template is not used, clean the template between samples to avoid cross-contamination and provide the laboratory with a blank wipe of the cleaned template between samples to ensure that no cross-contamination has occurred.

The instruction presumes that the samples will be collected from flat surface to which one may actually secure a template which neatly fits over the surface. In reality most of the surfaces that are sampled do not fit into this category and are very often (if not usually) convoluted surfaces to which a template cannot be applied; example include chandeliers, hanging lighting fixtures, spherical lamp covers, ceiling fan motors, tools, kitchenware, curtain rails, Venetian blinds, computer keyboards, construction materials (hangers, cleets, etc), fan blades, interior duct vanes, etc.)

Recommendation:
The language should be removed, and substituted with something such as:
6. Having selected the appropriate surface to be sampled, the surface area should be measured or estimated.
(In our case, we do not even submit the size of the area sampled and we require the laboratory to exclusively report mass of analyte recovered.)

Response 4: NIOSH Methods are written for the analyst as well as the IH. As mentioned above in a previous comment, the directions for measuring the surface area will be modified to accommodated surfaces that are not flat.

5. SAMPLING:

8. Cap shipping containers securely and keep refrigerated (<6 °C).
Recommendation:
This sentence will result in virtually all samples collected being challenged and possibly rejected. In forensic work, such a statement will be used to invalidate every set of samples since the requirement is both virtually impossible to ensure and, to my knowledge has no factual basis for support. This sentence alone should be sufficient for a forensic investigator to reject the entire method and use their own ad hoc method, and when on the stand asked why standard protocol wasn't used, the investigator would point to this recommendation and explain that the method cannot feasibly be followed.

Furthermore, the statement contradicts the last statement in the paragraph which reads:
...refrigeration is recommended as soon as possible (see Table 5).
I recommend that the language be rewritten thusly:

8. *Cap shipping containers securely and keep away from excessive heat and light.*

Response 5: NIOSH is not in the business of speculating on legal issues, which are for the courts to decide, not for NIOSH to weigh in on. The chemistry and validity of the method is our area of expertise, but analytical chemistry and the law are two completely different things. The wording for shipping samples

will be modified to advise the IH to follow proper chain-of-custody procedures[2] and to suggest that shipping samples cold is good practice.

6. *SAMPLING:*
(8) Containers must have no chips, fractures, or other irregularities on the sealing edge.

Recommendation:
I do not know what this means. Perhaps some clarification is needed.

Response 6: Wording in the method will change to convey that the containers need to be tightly sealed. Use of hard-walled, sealable containers, as elucidated in, e.g., ASTM D6966, is to be recommended.

Comment 7:
SAMPLING:

9. Label each sample clearly with a unique sample identification number or name, and the date, time, location, and initials or identification number of the individual taking the sample. The above information and a description of the sample and the area wiped should also be recorded in a logbook for later correlation with the analytical results.

Recommendation:

The following language should be added:

Sample identifiers shall not contain any QA/QC information such as "Blank," "duplicate," "spike" or any other identifier that indicates the nature of the sample. The sample should not contain specific location of the sample. Each sample should bear just a sample identification number, and the laboratory submittal sheet should bear exclusively a sample identifier and the size of the area wiped.

Response 7: The labeling of field samples is a standard procedure for an industrial hygienist and not something that is spelled out in great detail in each NMAM method. No changes to the method will be made in response to this comment.

8. SAMPLING:

10. Prepare a minimum of two field blanks with one field blank for every ten samples originating from the same clandestine laboratory or location.

Recommendation:

Although the more QA/QC one can employ is ideal, in the real world, this will be rejected and used by opposing counsel to invalidate the data set. In truth, those who will be purchasing the sampling will balk at the collection of unnecessary QA/QC and the forensic investigator would be at a loss to explain on the stand why two blanks were necessary for every ten samples. Furthermore, blanks are necessary to make a QA statement about the sampling materials and handling, not specific methlabs. Therefore, if say, a sample suite of say three labs were processed in one day; the investigator has prepared the sampling materials in a clean off-site location. Thirty samples are to be collected (three from one lab, two from on lab and 25 from the third lab). Three blanks

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[2] e.g., ASTM D4840, *Standard Guide for Sampling Chain-of-Custody Procedures.* ASTM International: West Conshohocken, PA, 2010.

would be adequate for the sample suite, since the three blanks will have been prepared and handled exactly as the remaining samples, and indeed, even the investigator will not know which samples ultimately will be identified as blanks, until the sample are actually laid out on scene. A blank frequency of greater than 10% cannot be justified outside of some other site-specific DQOs.

Response 8: NMAM methods are written in a standardized format. NMAM methods can be modified to fit the needs of the IH or analytical lab as long care is taken to check to make sure that any changes to the method are evaluated appropriately. The number of field blanks is a recommended amount. I would disagree with this reviewer about "field blanks" in that at least one field blank is recommended for each set or site where samples are taken. The blank media that are submitted to the lab for QA/QC, as long as they are from the same lot of media, can be used for analysis of all samples taken at the same time, but perhaps different locations. Proper QA/QC is well established in standardized methods, and minimum requirements are all that need be specified concerning QA/QC samples.

9. SAMPLING:

11. At least 3 laboratory media blanks are prepared at the rate of one for every 10 samples. Cotton gauze (unopened) from the same lot used for taking samples in the field should be provided to the analytical laboratory for preparing these laboratory blanks

Recommendation:

In the real world, this requirement would be ignored for several reasons. The first reason is that ultimately, the requirement results in a media blank frequency overkill. Most field assessments are fewer than 10 samples, (some 25% of assessments are only 2 samples, which are five-parted composites), and the proposed method would result in five blanks for just two samples.

Most sampling assessments in methlabs are performed for about $500, and that is a burden for the most common customer – a homeowner. To increase the cost, by increasing the blank frequency, without justification, would result in the method being not used by anyone.

If the field blanks (which ARE media blanks) are properly prepared, the media blanks at a rate of 10% are quite adequate. I have collected well over 1,300 samples, and our media blank log clearly indicates that 10% blank frequency is adequate (on only one occasion, have we seen detectable methamphetamine in a field blank, and we tracked that down to a laboratory error).

Response 9: See response 8.

10. ***SAMPLING:***

14. DESORPTION FROM MEDIA:

Recommendation:

In many cases, the investigator has used a shipping container that permits the extraction process to take place in the shipping container itself.

Response 10: Noted; no change will be made to the procedure, as the method has not been evaluated for extraction to take place in the shipping container.

11. ***Table 2:***

Table 2 contains some misinformation regarding Colorado's contamination limits. Contrary to erroneous statements frequently found in some literature, the value of "0.5

µg/100cm2" is not the State of Colorado cleanup level, but rather is the value upon which the final cleanup level is based and which is described in the mandatory Appendix A of the State regulations. The Colorado clearance level of "0.5 µg/100cm2," frequently misquoted by members of the general public, applies exclusively as *prima facie* evidence of decontamination at the end of a project[3] and is that attainment threshold occasionally needed to issue a "decision statement" (final clearance). Under those circumstances, the clean-up level becomes 0.5 µg/100cm2 divided by the number of samples in the wipe, up to five samples. Therefore, for a single discreet sample location, the limit is 0.5 µg/100cm2, however for a five parted composite, the limit is 0.1 µg/100cm2.

Response 11: The wording in Table 2 will be updated to show the current regulatory levels.

Comment 12: Contrary to popular misconception, there is **no** *de minimis* concentration during a Preliminary Assessment or a cursory evaluation below which a property could be declared "not a meth lab" or "not of regulatory concern" since virtually any concentration of meth present in a sample at the property would:
…lead a reasonable person, trained in aspects of methamphetamine laboratories, to conclude the presence of methamphetamine, its precursors as related to processing, or waste products.[4]

Therefore if, during an assessment of a property, an Industrial Hygienist collected five samples, from the property, and reported the following:
0.001 µg/100cm^2
0.002 µg/100cm^2
0.001 µg/100cm^2
<0.001 µg/100cm^2
0.001 µg/100cm^2
The data **CANNOT** be used to indicate the property is below regulatory limits. According to State regulations, the sample results MUST exclusively be used to trigger the need for Preliminary Assessment (which in this case would almost certainly result in a Decision Statement releasing the property).

Also, when I prepared the original language for the Colorado regulations, I specifically included MDMA, ephedrine, and pseudo ephedrine. According to Colorado State regulations:

"Methamphetamine" means dextro-methamphetamine, levo-methamphetamine, and unidentified isomers of the same, any racemic mixture of dexto/levo methamphetamine, or any mixture of unidentified isomers of methamphetamine. The term includes derivatives, conjugates, oxides,and reduced forms of the basic structure associated with CAS registration number 537-46-2. For the purposes of this regulation, this term also includes amphetamine (CAS 300-62-9), ephedrine (CAS 299-42-3), and pseudoephedrine (CAS 90-82-4).

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[3] Colorado Department Of Public Health And Environment, State Board Of Health, *Regulations Pertaining to the Cleanup of Methamphetamine Laboratories,* 6 CCR 1014-3.

[4] *Ibid.*

Response 12: The comment is not understood; in any case, what, if anything, is being asked of the editors here? NIOSH Occupational Exposure Limits and Standards are set on occupational exposures. The analytical method has been developed so that it will be fit for purpose.

Comment 13:

Appendix

Composite Samples:

We do not necessarily accept the "Composite sample" discussion, but rather, in the interests of expediency, pass comment on this section. If requested, we will review the discussion in depth.

Response 13: No response needed. NIOSH IH's generally do not use composite samples; it depends on the questions being asked. Do you need a lower detection limit or save money with fewer samples. Typically, composite samples are deprecated because of the analytical problems they often cause.

Comment 13: Field Duplicates

We disagree with the recommendations on collection of field duplicates since the distribution of contamination can be vast, even over very small distances.

Field duplicates are useful for evaluating the consistency of sampling technique, assuming uniformity of contamination on adjacent sampling sites

The statement incorporates a poor assumption. As such, the field duplicate should be collected by selecting an area to be sampled and dividing the area into even columns. The area is wiped in the normal fashion; each alternating column is assigned to a single sample identification.

Response 13: This statement will be deleted. NIOSH IH's generally do not do duplicate sampling for wipe samples.

External Reviewer:

EXTERNAL REVIEWER #2

Comment 1: My comments on the method itself are suggestions for consideration, I believe the method can be published as-is but these suggestions may make the method easier to follow.

1) On the first page the "SHIPMENT:" requirements do not include the use of a cooler and ice although several areas within the method suggest refrigeration of wipe samples soon after collection. I think the method should be written to include shipping samples (or transporting samples) in a cooler with bagged ice and custody seals to be consistent with the recommendation to refrigerate and protect samples from possible tampering found in the supporting documents.

Response 1: The change was made as indicated.

2) Page 2 of 32 in "SOLUTIONS:" step 6 – Why not write "0.3N hydrochloric acid in methanol: Dilute 2.5 mL conc. hydrochloric acid in 97.5 mL methanol." Or "0.3N hydrochloric acid in methanol: Dilute 2.5 mL conc. hydrochloric acid in enough methanol to make 100mL."

Response 2: Changes made as indicated.

3) Page 4 of 32 - #10 – The first sentence is confusing – "…minimum of two field blanks with one field blank for every ten samples…" this implies that more than ten samples

are always collected – many labs have fewer than ten samples collected from it – suggest a specific collection rate. Also, a field blank sample rate of 10% seems excessive. Consider suggesting "…no less than 5%, or one per batch, or 1 for each set of different equipment used (one for each lot of solvent and wipes)."

Response 3: Language will be clarified as suggested.

4) Page 10 of 32 Second sentence of the 3rd paragraph: "A second precision and accuracy test using methanol confirmed that methanol was an acceptable substitute." Does this mean that long and short-term sample stability is roughly the same for isopropanol and methanol? That a wipe sample collected with methanol is stable for up to 7 days without refrigeration? If this has not been determined for methanol then emphasizing sample refrigeration is potentially even more important for methanol collected samples (which is likely going to be most of the samples collected due to its increased sample accuracy).

Response 4: Language will be clarified as suggested.

5) Page 16 of 32 – Table 5: This table does not indicate which solvent was used along with the cotton gauze and whether substituting with another solvent changes the stability.

Response 5: The back-up report will be checked and the Table will be clarified as to which solvent is used.

External Reviewer:

EXTERNAL REVIEWER #5

Comment: The SIM tuning and data acquisition requirements are not specified. Presently, instruments may be tuned in any manner at the discretion of the laboratory, and may include tuning to Scan mode requirements with an accompanying loss in sensitivity. I have observed that the tuning algorithms proposed are designed to maximize the 69 atomic mass unit ("amu") ion for the tuning compound perfluorotributylamine ("PFTBA") inherently produce a better signal to noise ratio, and a lower detection limit. These tuning algorithms are typically referred to as the "Autotune" instrument option. The proposed tuning specifications are ambiguous, and may produce ambiguous data. In analyzing data produced from these methods, the agency will require that tuning be accomplished by way of Autotune protocols, and the following conditions must be met: (1) The operator must confirm that the 69/70, 219/220, and 502/503 isotope ratios occur at the proper ratios of 1 percent (+/- 50 %), 5 percent (+/-25 %), 10 percent (+/- 10 %) respectively.; (2) The peak width at half height for the 502, 219, and 69 PFTBA isotopes be 0.5 amu +/- 0.2 amu: and (3) The operator must confirm the correct mass assignment of these isotopes to a tolerance of 0.1 amu (e.g., 69.0 amu +/- 0.1 amu).

Once tuned, these instruments have acceptable electronic drift; such that, operators must verify that the tuning is stable at a minimum of once per operating day to insure correct mass axis alignment, and eliminate data accumulated with contaminated ion sources. These instrument tuning requirements specify the minimum acceptable performance goals which are easily verified.

I observed an apparent typographic errors in the existing tuning requirements, Suggested Tuning Requirements for the Scan Mode, Table 7 each method (9106/9109):

- m/z 119 should be m/z 219

Response: Correction made.

- The Scan Rate at 2 /second is not performance based. Consistant with other specifications, the agency will require a minimum of 10 scans across a peak, and this scan rate may need to be adjusted on certain instruments.

Both of these methods, in Table 8, footnote (3) proposes a dwell time of 50 milliseconds. It is clear that this suggestion does not optimize the data acquisition for maximum sensitivity, nor will this recommendation meet a minimum of 10 scans across the peak. This acquisition parameter must be adjusted to optimize the response.

In tabular form these are the minimum performance specifications for the use of this method in Colorado in support of agency projects:

Minimal acceptable requirement for analysis of wipe samples

Activity	**Specifications**	**Documentation needed**
GC/MS Tuning	Autotune or equivalent.	Printout of tune report.
	Acceptable Isotopic ratios (1, 5, 10 %)	
	Peak width at half height (0.5 amu +/- 0.2)	
	Correct mass assignment (+/- 0.1 amu)	
	10 scans across a peak	Demonstration.

Confirmed Positive detections: (REPORT: Concentration, qualify quantitative estimates with a "J")

- ion relative retention time tracks that of standards ***(+/- 0.10 RRT)***
- characteristic ion abundance ratio tracks ratio of standards (+/- 25 %)
- characteristic ions maximize within +/- one scan

Unconfirmed detections: (REPORT: Detected not confirmed, specify reason. Qualify quantitative estimates with a "J")

- ion relative retention time tracks that of standards ***(+/- 0.10 RRT)***
- characteristic ion abundance ratio fails to track ratio of standards (+/- 25 %)
- characteristic ions do not maximize within +/- one scan

Response: The methods that are developed for inclusion in the NMAM do not have performance based specifications. It is assumed that the analyst for this method is familiar with the analytical technical which for this method it is mass spectrometry. Typically the NMAM assumes that the analyst would do standard checks of the instrument to make sure it is performing to the Instruments specifications. This discussion isn't appropriate for the method but could be included in the backup data report.