Response to Outside Review of NMAM Method 9109 (Methamphetamine and Illicit Drugs, Precursors, and Adulterants on Wipes by Solid Phase Extraction)

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

Editors response to reviewers are in red.

Reviewer:

Comments – Single blind independent laboratory evaluation
reviewed the single blind independent laboratory evaluation of NIOSH 9109 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. A minimal amount of MDMA (between the Limit of Detection (LOD) and the Limit of Quantitation (LOQ)) was 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 reviewer's report will be added to the method or backup data report as a reference.

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.

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[1] ASTM D7144, Practice for collection of surface dust using micro-vacuum sampling; Creek, K., et al., J. Environ. Monit. 6: 612-618 (2006).

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**

Comment: There is a table in both the Backup Data Report (Table IV) 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 to the table in the method and the backup report will be made as indicated by the reviewer.

External Reviewer: EXTERNAL REVIEWER #2

Comment: 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.

Response 1: Changes made as indicated.

2) Page 2 of 33 in "SOLUTIONS:" step 8 – 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 33 - #10 – 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: This is a standard wording for the number of field blanks. No changes made.

4) Page 16 of 33 – 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 4: Changes made to clarify information in Table 5.

5) Backup Data Report for Method 9109
Page 5 of 82: Last sentence of first paragraph has 2 typos – "wsip" should be "wipe" and "prcedures" should be "procedures".

Response 5: Spelling corrected. Changes made as indicated.

External Reviewer: **EXTERNAL REVIEWER #4**

Comment 1:

SAMPLE AREA: 100 cm2 or 1 ft2 (929 cm2) as required by legal jurisdiction.

This statement creates several problems since:

It presumes there is a legal jurisdiction.
It presumes that 100 cm2 or 1 ft2 (929 cm2) is a norm.
It presumes that all sampling is performed under some regulatory requirement.
It propagates a myth that there is something "magical" about 100 cm2.

In Colorado, it would necessarily force an investigator to trigger a property for regulatory inclusion, where in fact, the property may not otherwise be included. For example, in Colorado, contrary to common misconceptions, there is no *de minimis* concentration during an initial assessment below which a property could be declared "not a meth lab" or "not of regulatory concern" or even "in compliance" 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.*[2]

So for example, if initial testing is conducted pursuant to Colorado's Real Estate transaction regulations, CRS §38-35.7-103, the Industrial Hygienist walks a fine line between ensuring that if toxicologically significant or regulatorily significant concentrations exist in the house, one has an high probability of identifying that condition; whilst at the same time, ensuring that one does not trigger the regulations for a property that may have merely trace amounts of methamphetamine.

To strike that balance, we, at FACTs, adjust our data quality objectives such that the total sampling area collected is such that if meth is present at significant levels, we will find it, and if it is present at trace levels we will report it as "below detection limit." As such, our reportable limit becomes 0.09 µg/100cm2; this is due to the fact that Colorado's minimum cleanup limit is 0.1 µg/100cm2 (**not** 0.5 µg/100cm2 as usually reported). Since our analytical RQ is 0.03 µg, our total sampling area is 32 cm2.

Therefore, we recommend the following language:

> *SAMPLE AREA: As required by legal jurisdiction, or commensurate with site specific data quality objectives.*

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[2] *Ibid.*

Response 1: 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,[3] and language from that standard will be incorporated to allow for an alternative to template-assisted sampling.

Comment 2: On the other end of the spectrum, occasionally there is a need (regulatorily and investigative) for extremely low detection limits. For example, last week one of our samples covered 8,400 cm2. This is a legitimate sample area, commensurate with our DQOs, and there is nothing magical about 100 cm2 – however, since the language of the draft method states:

> *2. The following steps only summarize the overall sampling procedure and are not intended to be used as a shortcut or substitute for any additional requirements of a specific regulatory agency. However, there are three parameters that concern the wiping technique that are **essential** for this method (NIOSH 9109).*
>
> *1) Use 3" x 3" 12-ply cotton gauze (for 100 cm2 areas), or 4" x 4" 8-ply cotton gauze (for up to 1 ft2 areas).*

In truth, there is absolutely nothing essential about either the 3" x 3" 12-ply cotton gauze provision, the 100 cm2 areas provision, or 4" x 4" 8-ply cotton gauze provision or the 1 ft2 area provision. The danger with the language lies in the fact that opposing counsel (criminal or civil) will jump on that language and use it to invalidate otherwise valid samples and sampling.
For example, the above referenced sample which was collected from 8,400 cm2 was collected with a 2" X 2" Johnson & Johnson cotton gauze wipe prepared from a 20 foot roll of material. The sample surface was a very smooth, high-gloss painted ceiling and was properly collected, from an area that met our DQOs and analyzed in an appropriate manner. As a field investigator, I would find a standard method of immense value, but NISOH methods that do not reflect realities in field sampling or real life scenarios make work for me in that I then have to spend extra time in my reports explaining why methods such as the NIOSH 9109 are not realistic and therefore, not followed.

Response 2: NIOSH is not a regulatory agency and does not comment on legal issues.The method will be modified to have all legal language taken out. Wording on method requirements or equipment will allow for other acceptable or similar equipment.

Comment 3:
Similarly, although we actually use 50-mL screw-capped polypropylene centrifuge tubes, there is nothing *essential* about :

> *3) Shipping containers: use 50-mL screw-capped polypropylene centrifuge tubes*
> *…*

And there is similarly nothing essential about

> *… up to two gauze wipes*

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[3] 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.

or

and 100-mL wide-mouth bottles with Teflon® lined cap for up to 4 gauze wipes (composite samples).

Strong language such as "shall," "essential" or any allusion to the same inhibits the professional decision making process and ensures that the method will either not be used, and/or the data set will be challenged precisely because the consultant did (or did not) use the method correctly. In other words, as an rebuttal expert, I could easily impugn an opponent's data set precisely because he *did* follow the method, which would have required a deviation from good sampling theory for the site specific conditions.

Response 2: The wording in the method will be changed to "suggest" needed equipment or something comparable. Some mandatory language is necessary in the method and will be retained where appropriate.

Comment 3:

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 according to the dimensions required by the regulatory agency. The template must be able to retain its shape during wiping to ensure that the areas wiped were 100 cm2 or 1 ft2. Single-use disposable cardstock is preferred because it eliminates the possibility for cross-contamination and the necessity to take a blank wipe between samples in step 5. (8) ***

5. 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 laboratory with a blank wipe of the cleaned template between samples to ensure that no cross-contamination has occurred.

The practice of specifying rigid templates is becoming a myth that greatly restricting the selection of 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 sample collection 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.

In processing a methlab last week, we encountered where a law enforcement agency entered a property and collected dozens of samples – few of which were appropriate, but all of which were collected from surfaces exclusively because the surface allowed the placement of a template.

There is nothing inherently incorrect with collecting a sample from a surface and then measuring the area and recording the area. In fact, in many cases, (such as the collection of a sample location in an extremely hard to reach location), frequently the sample will be collected and a photograph of the area made – from the photograph, the actual surface area is estimated and recorded. Imagine for example, inside a crime scene property that has been heavily disturbed, and essentially wipes clean - however the bad guy forgot about the wet bar in the basement and there are lot number on the liquor and wine bottles that allow a time stamp for manufacturing, and each bottle has historical dust. Imagine attempting to lay out a template on the tops of five liquor bottles! Templates are, at best, merely a single tool. Virtually none of our samples are ever collected with rigid templates.

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: 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,[4] and language from that standard will be incorporated to allow for an alternative to template-assisted sampling.

Comment 4:

Sampling

4. Provide enough wipe media from the same lot to cover all required laboratory media blanks, field-equipment blanks, samples and sample duplicates, and quality control samples. 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.

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[4] 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.

The use of the word "sterile" should not be used in this context since the sterility of the material is not an issue.

Also, sampling material should be prepared off site in a clean location. All completed sampling assemblies are then brought onsite. Samples are laid out and each sample container is given a unique sample identifier. Then using some method of random selection, the field BXs and duplicates are identified, and segregated from the sampling suit. Samples and dups are collected. And all samples are submitted blind to the lab. In this manner, the investigator provides *…enough wipe media from the same lot to cover all required laboratory media blanks, field-equipment blanks, samples and sample duplicates, and quality control samples.*

Gauze, and other materials should not be submitted alone unless the analysis is required by separate, specified DQOs.

Response 4: The clean media is to be used to make up samples for blank corrections and recovery studies in the analytical lab.
The **field blank** is a different sample that the industrial hygienist does not need to identify to the lab, but will let 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 an internal lab check; such samples are typical in carrying out any standardized analytical methodology.

Comment 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.

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.

SAMPLING:

8. *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.*

Response 5: The wording for shipping samples will be modified to advise the IH to follow proper chain-of-custody procedures[5] and to suggest that shipping samples cold is good practice. *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 6:

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.

SAMPLING:

10. Prepare a minimum of one field-equipment blank for every ten samples (originating from the same clandestine laboratory or location), and at least one for every clandestine laboratory or location being evaluated.

This language conflicts with DRAFT Method 9106 which reads

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:

We have made comments on the DRAFT 9106 Method regarding BXs and we have repeated those comments here. The 10% frequency is adequate without specifying a minimum of two field BXs. The most common client is an homeowner, and an additional $40 unnecessarily spent on additional field BXs, is sufficient to change a

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

client's mind and not bother with the sampling at all. The net result is that the consultant will ignore the method and use professional judgment anyway. Therefore, far better to promulgate a standard method that will be practical and will be followed.

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. 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 BXs would be adequate for the sample suite, since the three BXs 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 BXs, 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.

> *(10) …Prepare field-equipment blanks off-site to avoid contamination from dust or vapors on-site.*

This is an excellent practice, and should remain.

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 BXs for just two samples.

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

If the field BXs (which ARE media blanks) are properly prepared, the media BXs 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).

SAMPLING:

> *11. A laboratory media blank (QB) is prepared at the rate of one for every 10 samples. Cotton gauze from the same lot used for taking samples in the field*

should be provided to the analytical laboratory for preparing these laboratory blanks.

It is not the role of the analyzing laboratory to ensure QA/QC control over the in-field consultant or his materials. It is the role of the in-field consultant to exercise control over his materials and it is the role of the analyzing laboratory to maintain control over their reagents and their handling procedures. As such, unless the field investigator prepares separate DQOs for the submission and analysis of sampling materials, all sampling material QA/QC is adequately handled by the appropriate use of filed BXs.

Response 6: 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 and appropriately validated. The number of field blanks is a recommended amount. We 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.

Comment 7:
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[6] 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 7: The wording in Table 2 will be updated to show the current regulatory levels at the time of publication of the method.

Comment 8:
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:

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

...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.[7]

Therefore if, during an assessment of a property, an Industrial Hygienist collected five samples, from the property, and reported the following:

0.001 µg/100cm2
0.002 µg/100cm2
0.001 µg/100cm2
<0.001 µg/100cm2
0.001 µg/100cm2

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).

Response 8: 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 9:
Appendix
SAMPLING:

(5) Each regulatory agency having legal jurisdiction over the contaminated site may require different but very specific off-site preparation and on-site sampling procedures. It is important to consult local regulatory agencies or departments of

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[7] *Ibid.*

health having legal jurisdiction over contaminated sites to determine specific sampling, quality control, analyses, and reporting requirements.

Recommendation:
The language should be changed to:

(5) Prior to sampling, it is important to establish data quality objectives (DQOs) to ensure the resulting data are tenable and meaningful. Consult with local regulatory agencies or departments of health having legal jurisdiction over contaminated sites to determine specific sampling, quality control, analyses, and reporting requirements. A regulatory agency having legal jurisdiction over the contaminated site may require different but very specific off-site preparation and on-site sampling procedures. Otherwise, the field investigator should consult with professionals with expertise on sampling theory and clandestine drug laboratory operations prior to performing sampling.

Comment 9: Since NIOSH is not a regulatory agency and does not speculate on legal issues, the method will be modified to have all "legalese"-like language taken out.

Comment 10: 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 10: No response needed. NIOSH IH's generally do not use composite samples; it depends on the questions being asked. For example, 'Do you need a lower detection limit or save money on analyses with fewer samples?' Typically, composite samples are deprecated because of the analytical problems they often cause.

Comment 11: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 11: This statement will be deleted. NIOSH IH's generally do not do duplicate sampling for wipe samples. Duplicate sampling of surface samples is known to result in highly variable measurements owing to high variability in surface loadings over short distances. Hence, taking of duplicate surface field samples is deprecated.

External Reviewer: EXTERNAL REVIEWER #5

Comment 1:
Methods 9106 and 9109
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
- 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 level of detail being specified here is not appropriate for inclusion in the method. Methods that are developed for inclusion in the NMAM generally do not have performance based specifications; extensive discussion on desired performance criteria for NMAM sampling and analytical methods is covered in the Kennedy et al. NMAM chapter and in Kennedy et al. [1995][8]. It is assumed that the analyst for this method is familiar with the analytical technical which for this method,which 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 detailed level of discussion is not appropriate for the method but could be included in the backup data report.

Comment 2:
This method, as written, suggests the concurrent use of MSTFA and MBHFBA as a combined derivatizing reagent scheme. This does not make sense to me. I think the intent was to promote either MSTFA, or MBHFBA since each reagent develops a unique derivative. Table 8 in this method includes suggested ions for Heptafluorobutyryl-tri methyl silyl derivatives. When I reconcile the suggested ions present in Table 8, I deduce the ions in the table refer to Heptafluorobutyryl derivates from the use of MBHFBA. There is not an equivalent table for Trimethyl-silyl derivatives produced from MSTFA.

Response 2: The wording in the method will be clarified concerning when to use the different derivatizing agents.

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[8] Kennedy ER, et al., NIOSH Publ. No. 95-117.

General comment: Much of the information in the original version of the draft method will be moved to an appendix (such as instrumental parameters as elucidated in this example). Efforts have been made to shorten the method write-up itself and to provide other detailed information in appendices and/or the backup data report.