Response to Outside Review of NMAM Method 9111 (**Methamphetamine on Wipes by LC-MS)**

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 9111 conducted by a NIOSH contract laboratory (Bureau Veritas). The evaluation used wipe samples spiked with methamphetamine and then subsequently analyzed for the methamphetamine. Only significant quantities of methamphetamine were measured on the wipes. determined that LODs, LOQs, and recoveries of methamphetamine were high but were 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 smooth 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 this reviewer 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 mentioned by this reviewer 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. These alternate ways of testing surfaces for methamphetamine will be discussed in the appendix as well as the backup data report.

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. Recovery from different surfaces is discussed in some detail in the backup data report.

**External Reviewer:**

**EXTERNAL REVIEWER #3**

Comment:
There is a table in both the Backup Data Report (Table I) and method (Table I) that provides maximum surface contamination limits. The units in both are expressed as "µ/area". The correct units to express are "µg/area."
Many laboratories have LCMSMS which adds additional specificity and sensitivity compared to single quadrupole MS detection. I suggest the report address whether it is okay to use MSMS forming unique precurser/product ions to monitor for quant and qual ion pairs. I suggest adding language to make it clear that it is acceptable to alter the acid modifier concentration (optimum signal will vary with different systems and the optimal acid concentration may differ) and acid itself (many systems perform better using formic acid). Optimum voltage and gas values for the MS system will be instrument dependant and provide limited use to the reader if the specific instrument used is not listed.

Response: Corrections to the table in the method and the backup report will be made as indicated by the reviewer.
Even though MS/MS was not part of this method development a sentence will be added that this is another analytical option.
The language in the method discussing the acid modifier concentrations will be clarified as the reviewer suggests. Specifications of the voltages for the mass spec will be edited to allow for the use of any appropriate mass spectrometer.

**Reviewer:**

**EXTERNAL REVIEWER #4**

Comment:
I glanced at Method 9111 (Methamphetamine on Wipes by Liquid Chromatography-Mass Spectrometry-SIM), which was also identified as Method 0911 (I'm not sure if one is a typo).

In this case, I also glanced at the DataChem Backup Data Report, and would like to make a comment about the nature of the Colorado Standard listed there. The DataChem report identifies Colorado's clean-up level of 0.5 µg/100 cm2 as a "feasibility-based" clean-up standard. In my mind a "feasibility-based" standard implies a standard that is generally recognized as inadequate, but technically feasible to achieve.

When we developed the level of 0.5 µg/100 cm2, we actually looked at body burden modeling at various levels of residual contamination and on various surfaces to determine if we could see evidence of significant risk of adverse physiological harm based on the little known tox info for chronic low doses available at the time. What we found was that at the proposed threshold levels, there did not appear to be evidence that would support a strong argument for elevated risk. So, while not exactly an health-based standard either, I think it could be at least described as a "risk-based" standard. Although I didn't perform an in depth review of the method, I think that many of the concerns I raised regarding the 9109 and 9106 methods would apply.
If asked again, I will provide a more timely review of the methods in the future.

Response: The wording regarding risk-based standards, in the back up report will be in agreement with NIOSH standards. Reviewer comments for NMAM methods 9106 and 9109 will be used as appropriate in the finalization of the NMAM method 9111.

**External Reviewer:** **EXTERNAL REVIEWER #2**

Olympia WA 98502

Comment:

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.
2) Page 1 of 11 – Consider rewording "OTHER EVALUATED WIPE METHODS:" to "OTHER EVALUATED WIPE ANALYSIS METHODS:"
3) Page 2 of 11 – "SAMPLING: #2" - This sentence sounds like an introductory statement instead of a step in the procedure – consider moving this to the area above "#1".
4) Pages 2 and 3 of 11 – "SAMPLING:" The numbering in this section is non-sequential.

Response:

1.) The front page will be changed to recommend that shipping samples refrigerated is preferable.
2.) On page 1 the wording of "Other WIPE Methods" will have the word "Analysis" added.
3.) The wording of Sampling Step 2 will be clarified or moved to step 1.
4.) The numbering of the Sampling section will be corrected.