Review Form

Please complete a separate Review Form for each Docket.

Which Docket is being reviewed? (please underline)

Titles: NIOSH Manual of Analytical Methods (NMAM)

- **9106: *Methamphetamine and Illicit Drugs, Precursors, and Adulterants on Wipes by Liquid-Liquid Extraction (NIOSH-176)***
- 9109: *Methamphetamine and Illicit Drugs, Precursors, and Adulterants on Wipes by Solid Phase Extraction (NIOSH-177)*
- 0911: *Methamphetamine on Wipes by Liquid Chromatography-Mass Spectrometry-SIM (NIOSH-178).*

Anticipated Publication: NIOSH Manual of Analytical Methods (NMAM), 5th Edition

Return by: September 30, 2009

Return to: Dr. W. Gregory Lotz, Director, Division of Applied Research and Technology, Mailstop R-2, NIOSH, 4676 Columbia Parkway, Cincinnati, OH 45226, or email at wlotz@cdc.gov.

	YES	NO (explain below)
1. Does the Backup Data Report explain the problem and summarize relevant literature adequately?	___	_X__
2. Is the information in the Method and Backup Data Report technically accurate?	___	_X__
3 Are there any recommendations concerning organization of the documents?	___	_X__
4. Are there any changes or corrections needed in the Backup Data Report?	_X__	___
5. Are there any changes needed in the Method?	__X_	___
6. In general, is the Method and Backup Data Report satisfactory?	_X__	___
7. What is your recommendation for this Method as now written? (Check One):		
a. Approve for publication/dissemination	___	___
b. Approve after modification (please describe)	___	___
c. Do not approve (please describe)	_X__	___

DETAILED COMMENTS: (Provide comments in this space or on a separate sheet. Check here _X__ if a separate sheet is attached).

1. We are not sure of which "problem" is being addressed. Please see our accompanying notes.

2. Please see attached notes.

3. No explanation required.

5. Please see attached notes.

REVIEW OF
DRAFT *Methamphetamine and Illicit Drugs, Precursors, and Adulterants on Wipes by Liquid-Liquid Extraction*
(NIOSH 9106)

The following comments are not peer reviewed and are informal observations regarding the method under discussion. In the interests of expediency, we have not critically reviewed the analytical section.

Statement of Competency

Extensive laboratory experience in US CLP laboratories, and commercial labs using various NIOSH methodologies. Limited experience in GCMS and no direct experience in analyzing methamphetamine or related compounds by GCMS.

Extensive experience in sampling (including clandestine drug lab sampling), developing DQOs, sampling theory and authoring regulatory language.

SOLUTIONS:

> *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 v other polar solvents been investigated?

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.

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.

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 presume 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 revocered.)

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:

> *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.*

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 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.

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 – 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 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 wee seen detectable methamphetamine in a field blank, and we tracked that down to a laboratory error).

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.

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 <u>not</u> 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 <u>at the end</u> of a project[1] and is that attainment threshold occasionally

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

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.

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.*[2]

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).*

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[2] *Ibid.*

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.

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.