REVIEW OF
Methamphetamine and Illicit Drugs, Precursors, and Adulterants on Wipes by Solid Phase Extraction
(DRAFT NIOSH 9109)

The following comments are not peer reviewed and are informal observations regarding the method under discussion.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.

Many of the commends made here are applicable to the review of DRAFT Method 9106, and many of the comments we made regarding 9106 are applicable here.

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. See Clandestine drug lab SOQ at http://www.forensic-applications.com/meth/SOQ.pdf

A copy of this reviewer's resume is included as an attachment. A full curriculum vitae is available upon request.

HEADER:

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.[1]

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.

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

[1] *Ibid.*

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.

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*

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.

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.

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.*

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.

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 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 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.

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[2] 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 <u>divided by the number of samples in the wipe, up to five samples</u>. 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.

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

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.*[3]

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).*

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

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[3] *Ibid.*

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.*

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.