Group:

Here is a rough draft of the composite sampling guidelines and discussion. The discussion is by no means complete or thorough, but rather represents my first-pass thoughts on the philosophy of the approach. I am in favor of the calculated AL as proposed by Gene Hook (Denver Environmental Health) during the last meeting of the group, and I have taken his suggestion and presented it in a more quantified fashion. I look forward to discussions with the working group to hammer out a useable protocol-
Caoimhín P. Connell

DRAFT
Clandestine Methamphetamine Laboratories
Sampling and Analysis

The following document has been prepared as part of an ongoing regulatory promulgation process. This document has not been peer reviewed.

Revision: Original Draft
Date: August, 19, 2004

Purpose
The purpose of this appendix is to provide guidance on reducing variability in the collection of samples in the characterization of contaminants at illegal drug laboratories.

Scope and Application
The protocols identified in this appendix apply to the clean-up activities for illegal drug laboratories as defined in CRS 25-18.5-103.

Sampling Theory
The type of sampling used for stationary structures and vehicles described in this protocol is a type of sampling recognized as "authoritative" sampling. Authoritative sampling is a nonstatistical sampling design that does not assign an equal probability of being sampled to all portions of the population. Industrial hygienists using this protocol will have *a priori* knowledge of the property to be sampled. The *a priori* knowledge, in the hands of a competent professional IH, permits immediate inclusion/exclusion of sampling areas, based on professional judgment. As such, the weight of validity of the data gathered with authoritative sampling is largely dependent on the knowledge and competency of the sampler.

With authoritative sampling, it is not possible to accurately estimate the concentration variance within a property as a whole. Also, due to its subjective nature, the use of authoritative sampling to demonstrate compliance with a regulatory standard is generally not advisable except in those cases that are anticipated (small volumes of waste and where contaminants in the property under study is either well above or well below the action level). The ASTM D 6311 recognizes two types of authoritative sampling: judgmental sampling and biased sampling; both of these sampling theories are used in this protocol.

Judgmental Sampling
The goal of judgmental sampling is to use process or site knowledge to choose one or more sampling locations to represent the "average" concentration within the context of the sampling area. Judgmental sampling designs can be extremely useful and cost-effective *if* the IH choosing the sampling locations has sufficient knowledge of the history of the clandestine laboratory under study. It is recognized that the sampling method is not entirely objective since the IH choosing the sampling locations could possibly intentionally distort the sampling by a prejudiced selection,

or if their knowledge in the clandestine lab in question is wanting. In those cases, judgmental sampling can lead to incorrect results being presented to the decision maker.

Bias Sampling

Biased sampling is the type of authoritative sampling that intends not to estimate average concentrations or typical properties, but to estimate "worst" or "best" cases (as described in ASTM D 6051-96). As described later in this protocol, the aim of the IH performing post-remediation sampling is to demonstrate the worst case scenario in the clandestine meth lab. The term "biased," as used here, refers to the collection of samples with expected high concentrations. For example, a sample taken at the source of the actual "cook," known release, spill or storage area could serve as an estimate of the "worst-case" concentration found in the functional space. This information would be useful in identifying the contaminant and estimating the maximum level of contamination likely to be encountered during a cleanup. Biased sampling, while having the ability to cost-effectively generate information, has similar philosophical disadvantages to that of judgmental sampling.

Establishing Hypothesis Testing

The foundation for the usefulness of any sampling protocol rests upon the establishment of appropriate data quality objectives (DQOs). Without such DQOs, sampling occurs in a vacuum and the strength of the results of the sampling may be extremely limited.

The DQOs are, in turn, driven by a thought process that proceeds from defining the problem, then quantifying the degree of the problem, defining what decisions are to be made based on the resulting data, and the degree of quality needed to ensure that the decision goals can be met. All sampling has error; all analysis has error. No realistic sampling and analysis protocol has a 100% guarantee of definitively characterizing any area or condition. Therefore, a realistic sampling and analysis protocol is one that minimizes error, and optimizes cost effectiveness, while increasing the probability that the DQOs will be met.

This sampling protocol begins with the end in mind; it is based on asking specific questions, and conducting sampling and analysis to answer those questions. In general, this protocol will rely heavily on maximizing the use of existing law enforcement, investigation, analytical and historical information (including process knowledge), thus reducing unnecessary, costly data-gathering activities, while at the same time ensuring that building occupants and the public are not placed at unnecessary risk. The protocol is not a substitute for professional judgment, but must be utilized by cognizant professionals in the application of their professional skills. Neither is the method a "cook-book" recipe that if followed, remediation is guaranteed, and risks are assumed to be zero. The evaluation of any specific area must necessarily be based on the totality of the circumstances.

This protocol has been divided into two distinct sets of DQOs; one for the preliminary (pre-remediation sampling) and one for the post-remediation sampling. The essential difference between the two lies in the hypotheses that are being tested.

Pre-remediation sampling

In pre-remediation sampling, the question that is being asked is "Is there evidence of the presence of methamphetamine production in this area?" The assumption (hypothesis) is that the area is clean i.e. "compliant," and data will be collected to find support for the hypothesis. Data (such as samples) are collected to "prove" the area is compliant. Sampling, if it is performed, is conducted in the areas potentially containing the highest possible concentrations of contaminants. Any data that disproves the hypothesis, including police records, visual clues of production, storage, or use or documentation of drug paraphernalia being present, is considered conclusive, and leads the decision maker to accept the null hypothesis and declare the area non-compliant. The strength of evidence needed to reject the hypothesis is low and is only that which would lead a reasonable person, trained in aspects of clandestine laboratories, to conclude the presence of methamphetamine, its precursors as related to processing, or waste products.

Post Remediation

In post remediation sampling, the question that is being asked is "Does this contain contaminants in excess of the regulatory standard?" The hypothesis is the area is non-compliant, and data is collected to test the hypothesis. In theory, the ability to prove the hypothesis necessarily becomes more difficult as the area becomes cleaner; and virtually impossible to prove in an area that is completely devoid of contamination. The lack of data supporting the hypothesis leads the decision maker to accept the null hypothesis and conclude that the area is compliant. Therefore, the role of the industrial hygienist in post remediation sampling, is not to demonstrate that the area is "clean," but rather, using bias sampling, to diligently attempt to prove, that the area is not clean. The strength of evidence needed to accept the null hypothesis is great; and failure to support the hypothesis results in confidence that risks have been greatly reduced.

Decision Statement

If, based on the totality of the circumstances, the decision maker finds that insufficient evidence exists to support the hypothesis that any given area is non-compliant, that area shall be deemed to be compliant with CRS 25-18.5-103 (2), and shall be released. If objective sampling data indicates contamination is less than the action level, that data may be used as *prima fascia* evidence that insufficient evidence exists to support the hypothesis that any given area is non-compliant.

Area Samples

Stationary Structures

Wipe Sample and/or Vacuum Sample

For clandestine laboratories, as defined in, 25-18.5-103, whose structural floor plan is not greater than 1,500 square feet, surface sampling shall be collected according to the following schedule. Exception: for pre-remediation scenarios, any and all other data may be used in lieu of sampling to reject the hypothesis.

For any given *functional space*, at least 500 cm2 of surface shall be sampled, unless the area is assumed to be non-compliant. At least 1,000 cm2 of total surface area must be sampled for any single clandestine laboratory identified pursuant to 25-18.5-103. An additional 100 cm2 must be sampled for every additional 500 square feet of structural floor space. No fewer than five samples shall be collected from any clandestine laboratory identified pursuant to 25-18.5-103.

The required area shall be composed of no fewer than five discreet samples or any number of samples greater than five, collected in accordance with the procedures outlined in the section on Composite Sampling. (insert rubric).

Surface wipe samples shall be collected according to the Wipe Sample Collection Protocol found in section (insert) of this protocol.

Vacuum samples (for carpeting and fabric materials) shall be collected according to the Vacuum Sample Collection Protocol found in section (insert) of this protocol.

Where the clandestine laboratory is located in a structure other than a single family dwelling, the potential of fugitive emissions must be considered, and each functional space must be deemed a separate clandestine laboratory. For example, if the clandestine laboratory was located in an hotel room, and evidence of contamination extended into the corridor, the elevator, the lobby, and one adjacent room; there would be four separate clandestine labs to evaluate: 1) The primary hotel room, 2) the corridor/elevator complex 3) the lobby, 4) the adjacent hotel room.

Each separate building exhibiting indicia of contamination shall be considered a separate clandestine laboratory. For example, where a single-family dwelling meets the definition of a

clandestine laboratory, and whose detached garage contains indicia consistent with 25-18.5-103, there are two clandestine labs; 1) the residence, 2) the detached garage.

Vehicles

Wipe Sample and/or Vacuum Sample

For clandestine laboratories, as defined in, 25-18.5-103, in vehicles, surface sampling shall be collected according to the following schedule. Exception: for pre-remediation scenarios, any and all other data may be used in lieu of sampling to demonstrate a non-compliant condition.

A minimum of 500 cm2 of surface shall be sampled, unless the area is assumed to be non-compliant. An additional 100 cm2 must be sampled for every 50 square feet of structural floor space for any RV, or camper. No fewer than three samples shall be collected from any clandestine laboratory identified in a vehicle.

The required sampled area shall be composed of no fewer than three discreet samples or any number of samples greater than five, collected in accordance with the procedures outlined in the section on Composite Sampling. (insert rubric).

Surface wipe samples shall be collected according to the Wipe Sample Collection Protocol found in section (insert) of this protocol.

Vacuum samples (for carpeting and fabric materials) shall be collected according to the Vacuum Sample Collection Protocol found in section (insert) of this protocol.

Outdoors

For clandestine laboratories with outdoor components, or clandestine laboratories which are exclusively outdoors, the following sampling shall be performed. (I recommend incorporating, by reference, the grid sampling method as described in the Midwest Research Institute's publication titled "Field Manual for Grid Sampling of PCB Spill Sites to Verify Cleanup" and referenced in 40 CFR §761.130) Samples shall be taken to a depth of no fewer than 4 cm and no greater than 8 cm. Sample volume should be at least 100 cm3 and no more than 250 cm3. (guidance on soil sampling can be found in ASTM D 5730, ASTM E 1727, ASTM D 4700, and the EPA EISOPQA Manual).

Composite Sampling

Composite sampling is permitted by this protocol. Any composite sampling must consist of like media, matrices or substrates. The mixing of media, matrices or substrates is not permitted. All individual samples (designated as *g*), from which any single composite is formed must be of equal volume (for liquids), equal surface area (for surface wipe sampling or vacuum sampling) or equal weight (for solids).

Composite Designs

Composite sampling can be implemented as part of a statistical sampling design, such as simple random sampling and/or systematic sampling. The choice of a sampling design will depend upon the specific conditions of the clandestine lab being assessed.

Simple Random Composite Sampling

Figure 1 below shows how composite sampling can be integrated into a simple random sampling design. In this figure, the sampled area could represent any surface or media about which a decision must be made (such as a series of walls, or carpeting or even contaminated soils). Randomly positioned field sample composites can themselves randomly grouped together into composite samples. The set of composite samples can then be used to estimate the mean and the variance of the results. Because the compositing process is a mechanical way of averaging out spatial variabilities, we assume[1] the resulting concentration data to be more normally

distributed than individual samples.[2] This is especially advantageous because the assumption of the statistical tests in this protocol is that the underlying data approximate a Gaussian distribution.

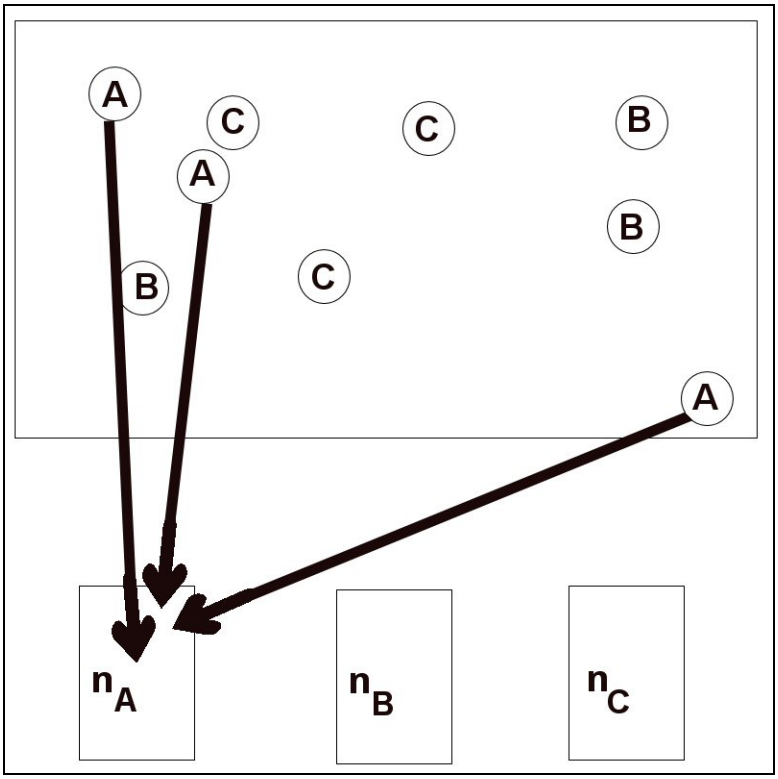
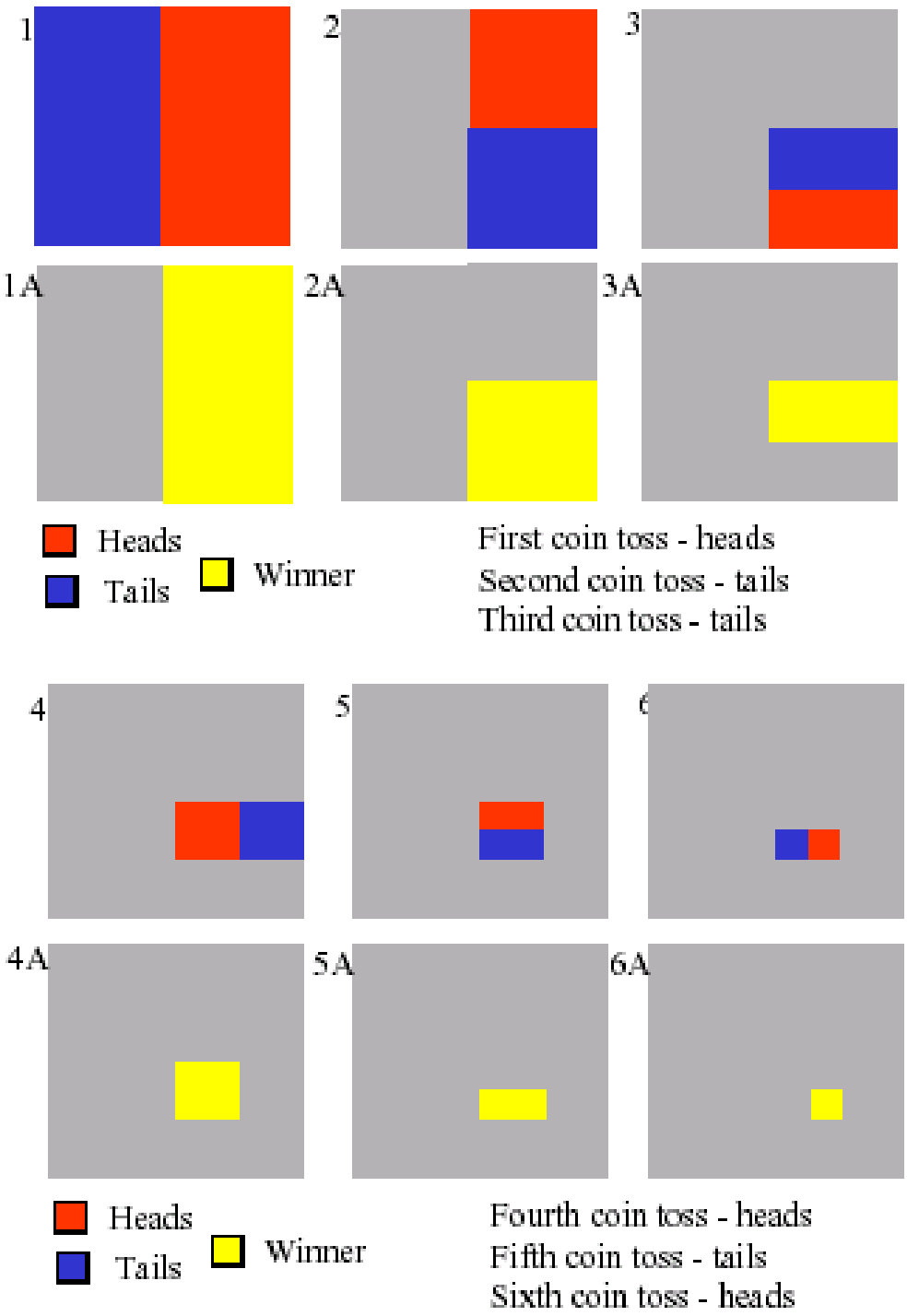


Figure 1
Example of Random Sample Composites

In the above example, nine individual samples (n*g=9) are composited into three samples for submission to a laboratory (X_A, X_B, X_C). Alternatively, the three composites may be combined to produce one sample submitted to the laboratory (X_{ABC}).

The sample locations can be selected by any number of methods. The "system of halves" as described in 40 CFR §761.306 is one example and is given below.

1. Select the surface which represents the area of highest possible contamination
2. Delineate one square meter encompassing the area
3. Divide the one square meter area in half with an imaginary line in any direction
4. Assign each half "heads" or "tails"
5. Flip a coin
6. Divide the "winning side" in half with an imaginary line in any direction
7. Flip a coin
8. Continue dividing the "winning" side until the winning side is between 100 cm2 and 200 cm2 and collect the wipe sample from that area
9. The method is repeated for each individual (*g*) of the composite



Systematic Composite Sampling

One kind of systematic composite sampling design is shown in Figure 2. The design can be used to estimate the mean concentration because each composite sample is formed from field samples obtained across the entire sampled unit (a wall, or a carpet, for example). Each field sample collected at the "A" locations is pooled and mixed into one composite sample. The process is then repeated for "B," "C," "D" locations and so on. The relative location and size of each individual field sample (such as "A") should be the same within each block.

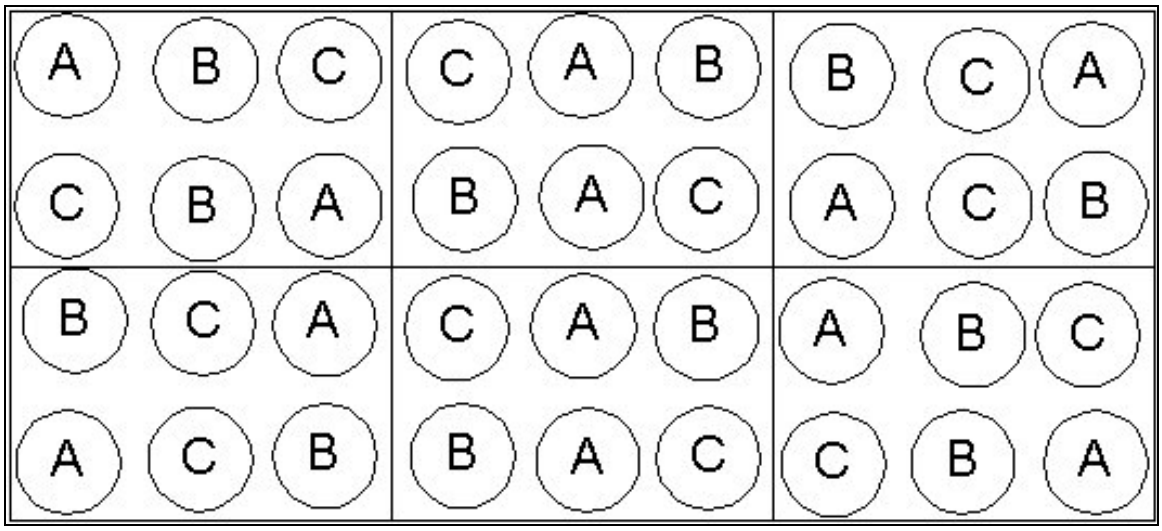


Figure 2
Example "A" of Systematic Sample Composites

A second type of systematic composite involves collecting and pooling samples from *within* a grid (See Figure 3).

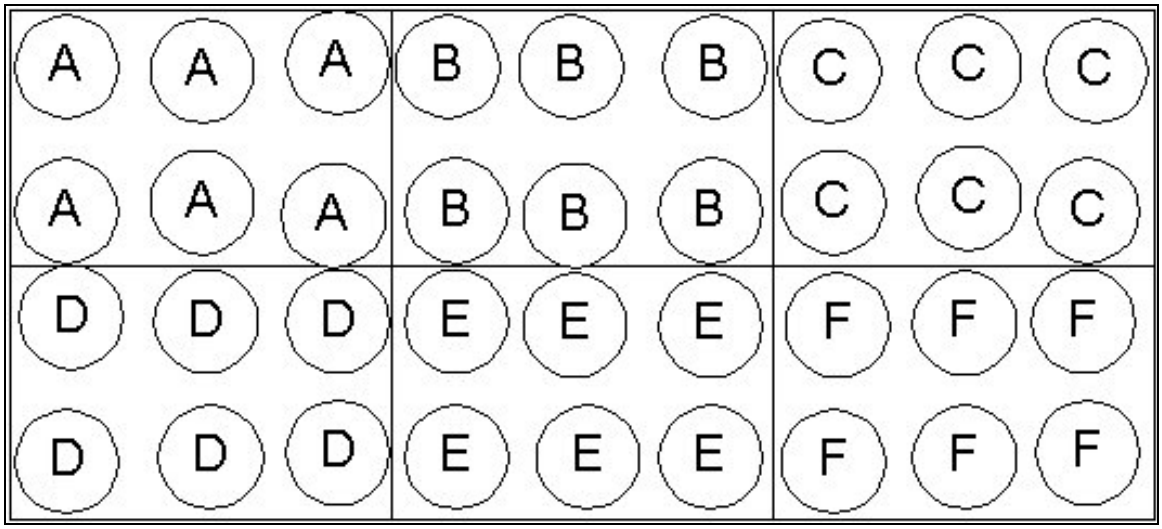


Figure 3
Example "B" of Systematic Sample Composites

If there is spatial correlation between the grid blocks, compositing within grids can be used to estimate block-to-block variability or improve the estimate of the mean within a block if multiple composite samples are collected within each block. In fact, compositing samples collected from localized areas is an effective means to control "short-range" (small-scale) heterogeneity. When this type of compositing is used on localized areas in lieu of "grab" sampling, it is an attractive option to improve representativeness of individual samples.

For post remediation, any of the above may be used, except, the industrial hygienist will purposely attempt to "high-grade" the samples (selectively choosing sample locations that represent the highest potential contamination, in accordance with the hypothesis being tested).

Composite Decision Level

One disadvantage of composite sampling is the possibility that one or more of the individual samples making up the composite could be "hot" (exceed the AL), but remain undetected due to "dilution" that results from the pooling process. If the sampling objective is to determine if any one or more individual samples is "hot," composite sampling can still be used.

The procedure for detecting hot spots using composite sampling is given below. The approach assumes the underlying distribution is normal and the composite samples were formed from equal-sized individual samples. Let AL be the "action level" that cannot be exceeded in any individual sample. AL is assumed to be large relative to the quantitation limit. For any laboratory result (X_i) from a composite sample formed from *g* individual samples, the following rules shall be assumed:

1) If $X_i < \frac{AL}{g}$ then no individual sample (*g*) shall be deemed greater than the AL

2) If $X_i > AL$ then at least one sample *must* be, and as many as all individual samples *may* be greater than the AL

3) If $X_i > \frac{AL}{g}$ then at least one of the individual samples (*g*) must be greater than the AL

4) As a general rule, no more than $\frac{g * X_i}{AL}$ individual samples can be greater than the AL.

(For example, if the laboratory reported 0.04 µg/100cm2, the maximum number of samples that composite could contain and still be compliant is six samples (*g*=6), since 6*g* = 0.48 µg/100cm2, and 7*g* = 0.56 µg/100 cm2.

If compositing is used then the number of samples that make up the composite should be limited to avoid overall dilution below the analytical limit. It is possible for a composite sample to be diluted to a concentration below the quantitation limit if many of the individual samples have concentrations near zero and a single individual sample has a concentration just above the action level. The maximum number of identically sized individual samples (*g*) that can be used to form a composite shall not exceed the action level (AL) divided by the quantitation limit (QL). As a practical matter, the number of individual samples used to form a composite should be between 2 and 10 discreet samples of equal area.

Regulatory Authority

Reserved

Glossary

action level:

the numerical value that causes the decision maker to choose one of the alternative actions (e.g., hypothesis or null hypothesis). For the purposes of this protocol, the action level for surfaces is established as 0.5 micrograms of methamphetamine per 100 square centimeters of sampled surface (0.5 µg/100cm2) for any discrete sample. For the purposes of the protocol, it is assumed that the analytical limit of quantification is less than the action level. The decision level for composite wipe and vacuum samples shall be calculated as described in Section (insert) Composite Decision Level. The action level for soils is established as (insert µg methamphetamine /g soil)

bias:

the systematic or persistent distortion of a measurement process which causes errors in one direction (i.e., the expected sample measurement is different than the sample's true value).

boundaries:

the spatial conditions and practical constraints under which environmental data are collected. Boundaries specify the area (spatial boundary) to which the decision will apply. Samples are then collected within these boundaries.

contaminant:

methamphetamine, its precursors, byproducts of processing, catalysts, and/or waste products.

Data Quality Objectives (DQOs):

qualitative and quantitative statements derived from the DQO Process that clarify assessment objectives, define the appropriate type of data, and specify the tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions.

Data Quality Objectives Process:

a Quality Management tool based on the Scientific Method to facilitate the planning of environmental data collection activities. The DQO Process enables planners to focus their planning efforts by specifying the intended use of the data (the decision), the decision criteria (action level) and the decision maker's tolerable decision error rates. The products of the DQO Process are the DQOs.

decision level:

that concentration, relative to the Action Level, that shall be used to distinguish between compliant and non-compliant areas. The calculation for the decision level for composite samples is found in Section (insert) Composite Decision Level

decision maker:

The independent industrial hygienist in conjunction with "The Board" as defined in CRS 25-18.5-101, *Definitions*. The Board will reserve the authority to reject data presented by the industrial hygienist.

decision error:

an error made when drawing an inference from data in the context of hypothesis testing, such that variability or bias in the data mislead the decision maker to draw a conclusion that is inconsistent with the true or actual state of the population under study.

defensible:

the ability to withstand any reasonable challenge related to the veracity, integrity, or quality of the logical, technical, or scientific approach taken in a decision making process.

EPA EISOPQA:

Environmental Investigations Standard Operating Procedures and Quality Assurance Manual

functional space:

"functional space" is a term of art used to describe any area containing any indicia of contamination. The "functional space" may be a single room, group of rooms, (including crawl spaces, attics, or the space between a dropped ceiling and the floor or roof deck above), designated by a the industrial hygienist who, based on professional judgment considers the space to be functionally separate from adjoining areas.

***g*:**
any individual sample collected for submission for analysis, either as a discreet sample or as part of a composite sample.

gray region:
a range of values of the population parameter of interest (such as mean contaminant concentration) where the consequences of making a decision error are relatively minor. The gray region is bounded on one side by the action level.

hypothesis:
a tentative assumption made to draw out and test its logical or empirical consequences.

industrial hygienist:
means a person as defined in CRS 24-30-1402

limits on decision errors:
the tolerable decision error probabilities established by the decision maker. Potential economic, health, ecological, political, and social consequences of decision errors should be considered when setting the limits.

matrix:
the material into which a substrate is collected. Matrices would include isopropyl alcohol, methanol, and water, etc.

mean:
(i) a measure of central tendency of the population (population mean), or (ii) the arithmetic average of a set of values (sample mean).

measurement error:
the difference between the true or actual state and that which is reported from measurements.

medium:
the physical material onto which a subtrate is collected. Media would include cotton gauze, glass fibre filters, MCE membranes, etc.

methamphetamine:
for the purposes of this protocol, the term methamphetamine includes dextro-methamphetamine, levo-methamphetamine, unidentified isomers of the same, any racemic mixture of dextro/levo or 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 protocol, the term also includes amphetamine (CAS 300-62-9), ephedrine (CAS 299-42-3), and pseudoephedrine (CAS 90-82-4).

natural variability:
the variability that is inherent or natural to the media, objects, or people being studied.

null hypothesis:
the default alternative conclusion that must be adopted if insufficient data exists to support the hypothesis

population:
the total collection of objects, or media to be studied and from which a sample is to be drawn.

qualified laboratory:
any laboratory that has implemented a QMP germane to the analysis of methamphetamine and uses, as a primary standard, the analyte being reported in the calibration of its instruments according to the QMP. (Qualified laboratories are (?) accredited and participating members in good standing with the …AIHA? NIOSH?)

quality assurance (QA):
an integrated system of management activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service (e.g., environmental data) meets defined standards of quality with a stated level of confidence.

Quality Assurance Project Plan (QAPP):
a formal technical document containing the detailed QA, QC and other technical procedures for assuring the quality of environmental data prepared for data collection activity and approved prior to collecting the data.

Quality Control (QC):
the overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the decision maker.

Quality Management Plan (QMP):
a formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation protocols of the laboratory for ensuring quality.

sampling:
the process of obtaining representative samples and/or measurements of a subset of a population. Sampling is a model; inherent in sampling is error, known or unknown.

sampling design error:
the error due to observing only a limited number of the total possible values that make up the population being studied. It should be distinguished from errors due to imperfect selection; bias in response; and errors of observation, measurement, or recording, etc.

standard deviation:
the square root of the variance.

stationary structure:
a stationary structure is a “building” as that term is defined in CRS 18-4-101. Not included are “vehicles” as defined in CRS 42- 1-102.

substrate:
the material being collected. Substrates may include soils, water, painted surfaces, carpet debris, unidentified powders, dust, etc.

total study error:
the combination of sampling design error and measurement error.

variable:

the attribute of the environment that is indeterminant.

variance:

a measure of (i) the variability or dispersion in a population (population variance), or (ii) the sum of the squared deviations of the measurements about their mean divided by the degrees of freedom (sample variance).

vehicle:

Any object that meets the common-sense interpretation for that object, excluding RV campers, buses with a toilet and a galley, and mobile homes. However, for clarity, "vehicle" is any object as defined in CRS 42-1-102 (complete through the 2002 legislative session) in paragraphs (8), (13), (14), (17), (17.5), (38), (44), (54), (55), (56), (57), (58), (59), (60), (60.5), (61), (71), (84), (88), (89), (93), (98), (99), (105), (106), (108), (109), and (112). Except, any vehicle that would be considered a "building" as defined in 18-4-102, shall be deemed a stationary structure for the purposes of this protocol.

X_i:

the laboratory analysis result for any discreet or composite sample submitted for analysis.

References

The following documents were consulted and used in the preparation of this protocol.

GUIDANCE FOR THE DATA QUALITY OBJECTIVES PROCESS EPA QA/G-4 EPA/600/R-96/055 September 1994

RCRA Waste Sampling Draft Technical Guidance Planning, Implementation, and Assessment EPA530-D-02-002 August 2002

ASTM consensus standard D 6044-96

[1] Based on the central limit theorem which states that if a population is repeatedly sampled, the means of all the sampling events will tend to form a normal distribution, regardless of the shape of the underlying distribution.

[2] Exner JH, Keffer WD, Gilbert RO, Kinnison RR. A Sampling Strategy for Remedial Action at Hazardous Waste Sites: Clean-up Soil Contaminated by Tetrachlorodibenzo-p-Dioxin " Hazardous Waste & Hazardous Materials 2(2):503-21, 1985.