METHAMPHETAMINE on Wipes by Liquid Chromatography/Mass Spectrometry

9111

$C_6H_5{\cdot}CH_2{\cdot}CH(CH_3){\cdot}NH{\cdot}CH_3$ MW: 149.2 CAS: 537-46-2 RTECS: SH4910000

METHOD: 9111, Issue 1 **EVALUATION: Partial** **Issue 1: 17 October 2011**

U.S. regulatory OELs
OSHA or MSHA: None for surfaces
Other published OELs and guidelines
ACGIH, AIHA or NIOSH: None for surfaces
States: Table 1

PROPERTIES: White solid, MP 171 - 175 °C; VP 0.163 mm Hg @ 25 °C; pK_a = 9.87 @ 25 °C; Water Sol. (1.33 g/100 mL @ 25 °C). Log P = 2.07 (octanol-water partition coefficient).

SYNONYMS: (*S*)-*N*,α-Dimethylbenzeneethanamine; (*S*)-(+)-*N*,α-Dimethylphenethylamine: *d*-1-Phenyl-2-methylaminopropane. Methedrine; Desoxyn; chalk; crank; crystal; glass; ice; meth, speed; upper.

SAMPLING

SAMPLER: Wipe

SAMPLE AREA: 100 cm^2 or 1000 cm^2

SHIPMENT: Ship refrigerated preferably

SAMPLE STABILITY: At least 7 days at 22 °C
At least 30 days at <6 °C

FIELD BLANKS: 2 to 10 blanks per set

MEASUREMENT ACCURACY

RANGE STUDIED: Not Determined

BIAS: Not Determined

OVERALL PRECISION ($\hat{S}_{rT}$): Not Determined

ACCURACY: Not Determined

MEASUREMENT

TECHNIQUE: LIQUID CHROMATOGRAPHY/MASS SPECTROMETRY-SIM mode. See Table 2.

ANALYTE: Methamphetamine

DESORPTION: 0.1 M sulfuric acid

INJECTION VOLUME: 50 µL

MOBILE PHASE: A: 5/95 acetonitrile/water, 0.1% acetic acid
B: 95/5 acetonitrile/water, 0.1% acetic acid

COLUMN: 150 mm x 4.6 mm, 5 µm Zorbax Eclipse XDB C18 x or equivalent. Column temperature 40 °C.

GRADIENT: 100% A for one min, gradient to 100% B (9 min), hold 5 min, gradient to 100% A (2 min), hold 8 min. Flow rate is 0.50 mL/min

CALIBRATION: Media spiked standards to cover the range (See Table 3)

RANGE: 0.1 – 100 µg/sample

ESTIMATED LOD: 0.1 µg/sample

PRECISION ($\overline{S}_r$): 0.067 [1]

APPLICABILITY: For methamphetamine the range is approximately 0.1 to 100 µg/sample (sample = 100 cm^2).

INTERFERENCES: No chromatographic interferences detected.

OTHER WIPE METHODS: NIOSH 9106 uses liquid-liquid extraction and gas chromatography/mass spectrometry (GC/MS) to measure multiple drugs [2]. NIOSH 9109 uses solid-phase extraction and GC/MS to measure multiple drugs [3].

REAGENTS:

1. Methamphetamine.* 1 mg/mL in methanol. (Alltech part # 010013 or equivalent).
2. Methamphetamine-D_{14} 1 mg/mL in methanol. (Cerilliant part # M-093 or equivalent).
3. Solvents, residue free analytical grades:
   a. Isopropanol (IPA) *
   b. Acetic Acid *
   c. Acetonitrile *
   d. Methanol *
4. Concentrated sulfuric acid (AR or trace metals analysis grade).*
5. Purified gas: nitrogen for drying.
6. Deionized water (ASTM type II).

SOLUTIONS:

1. Prepare spiking solutions of target analyte and internal standard. Keep solutions refrigerated. Protect solutions from light.
   a. Target analyte spiking solutions are prepared by diluting the 1000 µg/mL methamphetamine stock solution to 200 µg/mL and 20 µg/mL each in methanol.
   b. Dilute 1 mL of 1000 µg/mL methamphetamine-D_{14} stock solution to 10 mL for a 100 µg/mL (0.1 µg/µL) solution.
2. Desorption solution: 0.1 M sulfuric acid. Add 22 mL conc. sulfuric acid to 4 L deionized water.
3. Prepare Mobile Phase A: 0.1% acetic acid, 5% acetonitrile in water.
4. Prepare Mobile Phase B: 0.1% acetic acid, 95% acetonitrile in water.

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Wipe, cotton gauze, 3" x 3" (7.6 cm x 7.6 cm) 12-ply or 4" x 4" (10.2 cm x 10.2 cm) 8-ply or equivalent.
2. Sample storage and shipping container: 50-mL polypropylene centrifuge tubes with caps or equivalent.
3. Liquid chromatograph/mass spectrometer, with integrator or computerized data collection system, and column.
4. LC autosampler vials, 2-mL, and caps.
5. Volumetric flasks: various sized flasks for making standards and spiking solutions. A 4-L bottle for making the desorption solution.
6. Liquid Transfer:
   a. Various microliter syringes for making and spiking standard solutions.
   b. Adjustable 10 to 50-mL desorption solution dispenser to fit 4-L bottle.
7. Forceps.
8. Latex or nitrile gloves. Avoid vinyl gloves. (See 9111-3, Sampling, Step 1, NOTE 2.)
9. Rotating mixer capable of 10-30 rpm.
10. Pasteur pipettes.
11. Template: 10 cm x 10 cm hole. Template made of relatively rigid disposable cardstock or sheet of Teflon®.
12. Filters: Ion Chromatography Acrodisc®, 25 mm syringe filter with 0.45 µm Supor® (PES) membrane. (Pall number 4585T or equivalent).
13. Ice or other cold media for shipping.

SPECIAL PRECAUTIONS: The solvents are flammable and have associated adverse health effects. Avoid breathing vapors. Avoid skin contact. Work should be performed in an adequate hood. Analysts must wear proper eye and hand protection (e.g. latex gloves) to prevent absorption of even small amounts of amines through the skin as well as for protection from the solvents and other reagents. Dissolving concentrated sulfuric acid in water is highly exothermic. Goggles must be worn.

Caution must also be exercised in the handling and analysis of samples. Clandestine drug labs may produce unknown and seriously toxic by-products.

SAMPLING:

See APPENDIX for special instructions on sampling.

1. Using a new pair of gloves, remove a gauze wipe from its protective package. Moisten the wipe with approximately 3 to 4 mL of methanol (or isopropanol).
   NOTE 1: Apply no more solvent than that needed to moisten approximately the central 80% of the area of the gauze wipe. Excess solvent may cause sample loss due to dripping from the wipe.
   NOTE 2: Do not use vinyl gloves due to the potential for leaching of phthalate plasticizers and contamination of the samples.
2. Place the template over the area to be sampled (may tape in place along outside edge of template). Wipe the surface to be sampled with firm pressure, using vertical S-strokes. Fold the exposed side of the pad in and wipe the area with horizontal S-strokes. Fold the pad once more and wipe the area again with vertical S-strokes.
3. Fold the pad, exposed side in, and place in shipping container and seal with cap.
   NOTE: Keep samples refrigerated (<6 °C). While methamphetamine and several related amines are stable on the recommended wipe media for at least 7 days at room temperature, refrigeration is recommended as soon as possible.
4. Clean the template before use for the next sample or use a new disposable template.
5. Label each sample clearly with a unique sample identifier.
6. Prepare a minimum of two field blanks with one field blank for every ten samples.
   NOTE: In addition, include at least 3 media blanks for the analytical laboratory to use for their purposes. The wipes used for the media blanks should be from the same lot as the field samples.

SAMPLE PREPARATION:

7. Desorption from media:
   a. Remove cap from shipping container. Sample media should fit loosely in the container. If not, rearrange media carefully with rinsed forceps or transfer to a larger container. If the sample media are transferred to a larger container, do not discard the original container. Samples may consist of more than one wipe. If this is the case, internal standard and desorption solution volumes may be adjusted accordingly.
   b. Spike exactly 50 µL of internal standard spiking solution onto each wipe sample.
   c. Add 30 mL desorption solution (0.1 M sulfuric acid). If the samples were transferred to a larger container, the original shipping container must be rinsed with the desorption solution first, shaken, and the rinsate decanted into the larger container.
   d. Cap securely and mix contents by inverting the tubes end over end on a rotary mixer at 10-30 rpm for at least one hour.
      NOTE 1: The desorption solution must percolate freely through the gauze wipes.
      NOTE 2: If there is reason to believe that the samples may be alkaline enough to overcome the acidity of the desorption solution (e.g. wipes of unpainted concrete or stucco surfaces), then the pH must be adjusted to about ≤ 4. See APPENDIX for instructions.
   e. Filter an aliquot of the sample through a 0.45 µm pore size Ion Chromatography Acrodisc® for analysis.
8. Transfer the filtered sample into a vial and cap.
9. Analyze samples, standards, blanks, and Quality Control samples (QCs) by LC-MS. (See MEASUREMENT, steps 13-15.)

CALIBRATION AND QUALITY CONTROL:

10. Determine retention time using the column and chromatographic conditions specified on page 9111-1.
11. Calibrate daily with at least six media spiked calibration standards and a blank.
    a. Prepare the target analyte spiking solutions. (See SOLUTIONS, 9111-2)
    b. Prepare calibration standards and media blanks in clean shipping containers (e.g. 50-mL polypropylene centrifuge tubes.)
    c. Spike a known volume of target analyte spiking solution into each calibration standard by spiking directly onto the media. Use the spiking volumes suggested in Table 3 to cover the desired range.
    d. Analyze these along with the field samples. (See MEASUREMENT, steps 13-15.)
12. Prepare matrix-spiked and matrix-spiked duplicate quality control samples (QC and QD).
    a. Cotton gauze from the same lot used for taking samples in the field should be provided to the analytical laboratory to prepare these matrix-spiked QC samples.
    b. The quality control samples (QC and QD) must be prepared independently at concentrations within the analytical range. (See Table 3 for applicable concentration ranges.)
    c. One quality control media blank (QB) must be included with each QC and QD pair.
    d. The quality control samples must be prepared at the rate of one set (QB, QC, and QD) per 20 samples or less.
    e. Transfer clean gauze wipes to new shipping containers.
       NOTE: If two gauze wipes were used for the majority of samples in an analytical set, use two clean gauze wipes for each QB, QC, and QD.
    f. Spike QC and QD with a known amount of target analyte as suggested in Table 3.
    g. Process quality control samples along with the calibration standards, blanks, and field samples through steps 7 and 8.
    h. Analyze these along with the calibration standards, blanks, and field samples. (See MEASUREMENT, steps 13-15.)

MEASUREMENT:

13. Analyze the calibration standards, quality control samples, blanks, and samples by LC-MS.
    a. Use the following suggested analytical sequence.
       i. Calibration standards.
       ii. Matrix spiked quality control samples (QC and QD), one set for every 20 samples or less.
       iii. A media blank (QB), one for every 20 samples or less.
       iv. Samples (up to 10) including one sample duplicate.
       v. A continuing calibration verification (CCV) standard consisting of one of the initial calibration standards.
       vi. A media blank.
    b. Set liquid chromatograph according to manufacturer's recommendations and to conditions listed previously.
    c. Set mass spectrometer to scan for ions 119, 150, and 164 in SIM mode. Further suggestions for MS conditions are listed in Table 2 but will vary for particular instruments and conditions. See Note 2 in Table 2.
    d. Inject 50 µL of the sample aliquot into liquid chromatograph.
    e. After analysis, the vials should be promptly recapped and refrigerated if further analysis is anticipated. Samples are stable refrigerated for at least seven days.
14. Using extracted ion current profiles for the primary (quantification) ions specific to methamphetamine and the internal standard, measure the LC peak area of each respective peak and compute relative peak areas by dividing the peak area of the analyte by the area of the internal

standard. Recommended primary (quantification) ions and internal standard ions are given in Table 2. Prepare a calibration graph (relative peak area vs. µg analyte per sample).

15. Samples from initial investigations of clandestine laboratories are likely to include highly contaminated samples. If sample results exceed the upper range of the calibration curve, the sample in the LC vial may be diluted with the sulfuric acid desorption solution and reanalyzed.

CALCULATIONS:

16. Determine the mass in µg/sample of methamphetamine found in the wipe samples and in the media blank from the calibration graph.
17. Calculate final concentration, C, of methamphetamine in µg/sample:

$$C = c\frac{V_1}{V_2} - b$$

Where:

c = concentration in sample (in µg/sample determined from the calibration curve)

$\frac{V_1}{V_2}$ = dilution factor, if applicable

V_1 = volume in µL of internal standard spiking solution used to spike samples.

V_2 = volume in µL of internal standard spiking solution used to spike the standards.

b = concentration in media blank (in µg/sample determined from the calibration curve).

18. Report concentration, *C'*, in µg per total area wiped (in cm^2) as follows:

$$C' = \frac{C}{A}$$

Where:

C = ug/sample (step 17).

A = Total area wiped in cm^2 per sample.

NOTE: For example, if the sample was a composite sample and the area was 400 cm^2, report results as µg/400 cm^2. In general, if the area wiped was greater than or less than 100 cm^2, do not convert value to µg/100 cm^2. To avoid confusion, report separately both µg/sample (C) and the total area wiped in cm^2 per sample (A) for both discrete and composite samples.

EVALUATION OF METHOD:

This method was evaluated for methamphetamine over a range of approximately 0.4 µg/sample to 17.8 µg/sample on cotton gauze. These concentration levels represent approximately 3 through 100 times the limit of quantitation (LOQ) level. Results are reported in the Backup Data Report for 9111s[1].

The limit of detection (LOD) and LOQ were determined by preparing a series of media spiked standards, desorbing in the sulfuric acid desorption solution and analyzing in the SIM mode. The LODs were estimated using the procedure of Burkart [4]. An LOD of less than 0.02 µg/sample for methamphetamine on wipes was achieved in the SIM mode. The LOD was set at 0.05 µg/sample and the LOQ at 0.15 µg/sample for method development purposes. Lower LODs can be achieved in practice by including calibration standards at lower concentration levels and with proper instrument

maintenance. The cleanliness and performance of the mass spectrometer must be maintained such that at a minimum of 0.1 µg/sample a signal of at least 5 to 10 times the baseline noise is achievable.

Precision and accuracy were determined by analyzing 6 replicates at each of 4 concentration levels (nominally 0.44, 1.8, 4.4, and 18 µg/sample). Accuracy was calculated using equations and methodology found in the NIOSH Technical Report "Guidelines for Air Sampling and Analytical Method Development and Evaluation" [5]. Using all data, method precision (S_r) was 0.06663. Accuracy was 20.7% and mean bias was -0.09753.

Long term sample storage stability was determined for periods up to 30 days under refrigeration (4 °C ± 2 °C) and for up to 7 days at room temperature (22-24 °C). Since long term storage measures only the viability of analytes on a particular media over time, this determination was not repeated for this particular method; the reader is directed to NIOSH 9106 [2] for more detail. All recoveries were found to be 93.5% or better.

Recovery of amphetamines from six different types of surfaces using cotton gauze was evaluated. The study and results are reported in NIOSH 9109 [3]. The practice of serial wiping (wiping the same surface area a second time with a second gauze wipe and combining both wipes as a single sample) was evaluated. Four solvents for wetting the gauze were tested (distilled water, 5% distilled white vinegar, isopropanol, and methanol). Six replicate samples were taken on a latex painted wall. Recovery and precision results are presented in the previously mentioned Backup Data Report. In summary, the effectiveness of the various solvents using a single wipe on a latex painted wall were as follows: water, 46% recovery; 5% distilled white vinegar, 55% recovery; isopropanol, 64% recovery and methanol, 87% recovery. Average recoveries with isopropanol from all the surfaces tested were greatly improved with a repeat (serial) wipe (11% improvement compared to only about 6% improvement with methanol). The serial wipe is added to the first wipe and constitutes a single sample.

REFERENCES:

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[4] Burkart JA [1986]. General procedures for limit of detection calculations in the industrial hygiene chemistry laboratory. Applied Industrial Hygiene 1(3):153-155, (1986).

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[6] Martyny JW. Decontamination of building materials contaminated with methamphetamine [http://health.utah.gov/meth/html/Decontamination/DecontaminationofBuildingMaterialsContaminatedwithMeth.pdf]. Date accessed: May, 2007.

[7] Martyny JW [2008]. Methamphetamine sampling variability on different surfaces using different solvents [http://health.utah.gov/meth/html/Decontamination/MethSamplingVariabilityonDifferentSurfaces.pdf] Date accessed: May, 2011.

METHOD DEVELOPMENT BY:

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TABLE 1. Methamphetamine Regulations by State (Jan 2008)*

State	Standard	State	Standard
Alaska**	0.1 µg/100 cm^2	Minnesota	0.1 µg/100 cm^2 (meth labs) , < 1.5 µg/100 cm^2 (meth use)
Arizona	0.1 µg/100 cm^2	Montana	0.5 µg/ft^2
Arkansas	0.1 µg/100 cm^2	New Mexico	1 µg/ft^2
California***	< 1.5 µg/100 cm^2	North Carolina	0.1 µg/100 cm^2
Colorado	0.5 µg/100 cm^2	Oregon	0.5 µg/ft^2
Connecticut	0.1 µg/100 cm^2	South Dakota	0.1 µg/100 cm^2
Hawaii	0.1 µg/100 cm^2	Tennessee	0.1 µg/100 cm^2
Idaho	0.1 µg/100 cm^2	Utah	0.1 µg/100 cm^2
Kentucky	0.1 µg/100 cm^2	Washington	<0.1 µg/100 cm^2

The following states have no standard: Alabama, Delaware, D.C., Florida, Georgia, Illinois, Indiana, Iowa, Kansas, Louisiana, Maine, Maryland, Massachusetts, Michigan, Mississippi, Missouri, Nebraska, Nevada, New Hampshire, New Jersey, New York, North Dakota, Ohio, Oklahoma, Pennsylvania, Rhode Island, South Carolina, Texas, Vermont, Virginia, West Virginia, Wisconsin, Wyoming.

* NIOSH has not established health-based or feasibility-based airborne Recommended Exposure Limits (RELs) or surface contamination guidelines for clandestine drug laboratories. State surface contamination limits are provided as an aid to those seeking additional information and does not constitute endorsement by NIOSH. The National Alliance for Model State Drug Laws (NAMSDL) website: http://www.namsdl.org/home.htm periodically summarizes state feasibility-based decontamination limits and proposed state legislative requirements and guidelines. However, state information is subject to change, and specific state's surface contamination limits, and other state decontamination requirements and guidelines should be obtained directly from each state.

** Guidance and Standards for Cleanup of Illegal Drug-Manufacturing Sites Revision 1 April 19, 2007 Alaska Department of Environmental Conservation, Spill Prevention and Response Division, Prevention and Emergency Response Program.http://www.dec.alaska.gov/spar/perp/methlab/druglab_guidance.pdf

*** In Oct 2009 House Bill 1489 was passed into law to incorporate the new standard as the state limit. All other states: Data source: http://health.utah.gov/meth/html/Resources/OtherStates/Nationalcomparison (downloaded April 2011).

Table 2. Suggested Mass Spectrometer SIM Conditions

Ionization Mode:	API-ES (Atmospheric Pressure Ionization –Electrospray)
Polarity:	Positive
Fragmentator:	100
Gain:	3.0 EMV
Actual Dwell:	294
SIM ions:	119 Methamphetamine confirmation ion
	150 Quantitation ion for methamphetamine
	164 Ion for methamphetamine-D_{14}
Spray Chamber:	(Optimize for the particular instrument in use.)
Gas Temperature:	200 °C
Drying Gas:	12.0 L/min
Nebulizer Pressure	50 psig

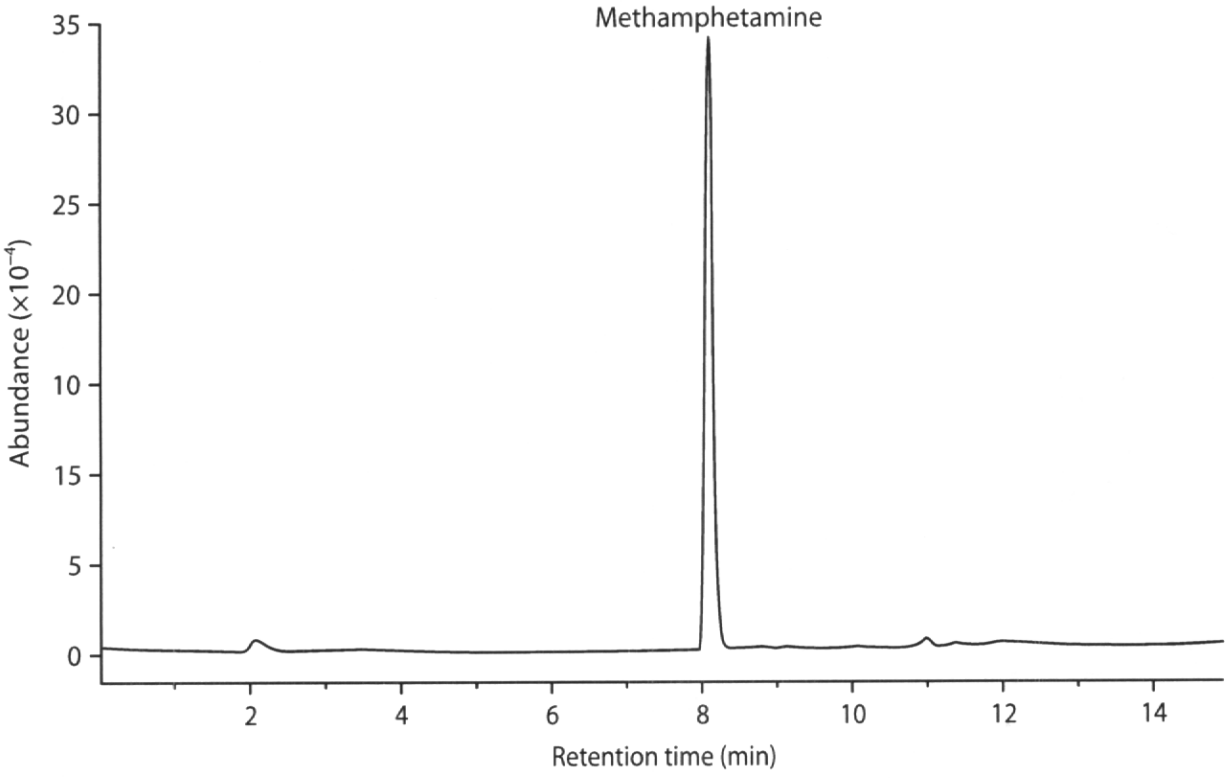
Note 1: Methamphetamine and the internal standard essentially co-elute. Monitor *m/z* ions 119 and 150 for methamphetamine quantitation and 164 for methamphetamine-D_{14}.

Note 2: These instrumental conditions are suggestions and should be optimized by the analyst. Each mass spectrometer will be different and the acid concentration and composition will alter the conditions. Furthermore there is the possibility of using MS/MS for these analyses if the laboratory is equipped with that instrumentation. This tandem MS could add specificity and sensitivity to the method but was not part of this method development.

Table 3. Suggested spiking schedule for calibration standards

	Volume (µL) of Methamphetamine Spiking Solution Spiked on Media				
Standard	200 µg/mL solution	20 µg/mL solution	Internal Standard Spike (µL)	Desorption Solution (mL)	Final Concentration (µg/sample)
1	500		50	30	100
2	100		50	30	20
3	25		50	30	5
4	5		50	30	1
5		25	50	30	0.5
6		5	50	30	0.1
7		2.5	50	30	0.05
8		1.2	50	30	0.024

Figure 1. LC-MS total ion chromatogram of methamphetamine (*m/z* 119, 150, 164).



APPENDIX:

SAMPLING

NOTE: For further information and data on the effects of surface composition and porosity as well as the use of different solvents on the efficacy of wipe sampling, please see reports by Martyny [6, 7].

1. Follow specific requirements of surface area to be wiped (usually 100 cm^2) and action threshold (or maximum allowable residual level) set by the agency having legal jurisdiction or specified by the client. Uptake rates depend upon the wipe sampling method used so the specific wipe technique used must be specified and any deviations from the required wipe sampling requirements noted.
2. The following steps only summarize the overall sampling procedure and are not intended to be used as a shortcut or wipe sampling procedures that may be specified by the legal jurisdiction or the client.
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 (1000 cm^2) 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 cm^2 or 1 ft^2. 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 3.
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.
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.
6. With freshly gloved hand, take one gauze and wet it with isopropanol or methanol (about 3-4 mL for either the 3" x 3" (7.5 cm x 7.5 cm) 12-ply or the 4" x 4" (10 cm x 10 cm) 8-ply cotton gauze wipes). Alternatively, pre-wet and insert the gauze wipes into the sample containers off-site. This avoids any possibility of the bottle of methanol or isopropanol becoming contaminated on-site with methamphetamine. If the wipes were prepared off-site, then remove pre-wetted gauze wipe from sample container, opening only one sample container at a time. In either case, squeeze out and discard any excess solvent from the gauze wipe. Use fresh latex or nitrile gloves for each separate sample and blank. Do not use vinyl gloves due to the potential for leaching of phthalate plasticizers and contamination of the samples.
7. Wipe Techniques
   a. Concentric Squares Wiping Technique (particularly suitable for smooth and non-porous surfaces): Fold the pre-wetted gauze in half and then fold in half again. Using firm pressure wipe the area within the template. Start at one of the inside corners of the template and wipe in concentric squares, progressing toward the center. End with a scooping motion. Without allowing the gauze to touch any other surface, reverse the last fold so that the exposed side of the gauze is facing inward and using a fresh surface of the gauze, wipe the same area in the same manner as before. Roll or fold the gauze again and insert into the shipping container.
   b. Side-to-side Wiping (or Blotting) Technique (particularly suitable for rough, porous, and/or soiled surfaces): Fold the pre-wetted gauze in half and then fold in half again. Using firm pressure wipe or blot the area within the template with at least five overlapping side-to-side horizontal passes (see NOTE) beginning at the top and progressing to the bottom in a "Z" pattern. End with a scooping motion. If blotting, blot at least five times on each horizontal pass (see NOTE). Without allowing the gauze to touch any other surface, reverse the last fold so that the exposed side of

the gauze is facing inward. Using a fresh surface of the gauze, wipe or blot the area again with at least five overlapping top-to-bottom vertical passes beginning at the left side and progressing to the right in an "N" pattern. If blotting, blot at least five times on each vertical pass. Roll or fold the gauze again and insert into the shipping container. Blotting is suggested in areas so soiled or rough that the threads of the gauze media are continually snagged.
NOTE: On areas larger than 100 cm^2, more than five passes and blots will be needed.

c. Repeat or Serial Wiping: If isopropanol is used for wiping, a serial or repeat wipe sample of the same area with a fresh gauze wipe will improve sampling efficiency. For serial wiping, repeat the wiping procedure described above (APPENDIX 7a or 7b) with a fresh gauze wipe. Place the second gauze wipe into the same shipping container as the first gauze. The 50-mL polypropylene centrifuge tubes are large enough to contain up to two gauze wipes of either the 3" x 3" 12-ply or 4" x 4" 8-ply sizes.
NOTE: If the area to be wiped remains substantially wet from the first gauze, the second gauze wipe might be used in the dry state to soak up the residual solvent from the first gauze wipe.

8. Cap shipping containers securely and keep refrigerated (<6 °C). Make sure caps are not cross-threaded. Containers must have no chips, fractures, or other irregularities on the sealing edge. Do not use polyethylene plastic bags. While methamphetamine and several related amines are stable on the recommended wipe media for at least 7 days at room temperature, refrigeration is recommended as soon as possible.
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.

SAMPLE PREPARATION

Samples requiring pH adjustment: If there is reason to believe that the samples may be alkaline enough to overcome the acidity of the desorption solution (e.g. wipes of unpainted concrete or stucco surfaces), then the pH must be adjusted to about ≤ 4. The pH may be checked with pH paper or monitored with the addition of about 2 drops of the mixed pH indicator solution of bromothymol blue and phenolphthalein. (The color should be yellow and not green or blue.) The preparation of this indicator solution can be found in NIOSH 9106 or NIOSH 9109. [2, 3] If the pH needs to be adjusted, a solution of dilute (2.5 to 3 M) sulfuric acid is used and added dropwise. Mix the contents by shaking or inversion a few times by hand after each addition of acid before checking the pH.