Clandestine Methamphetamine Manufacturing:

1) Methods of Meth Synthesis
2) Understanding Lab Reports and Chemical Evidence Lists
3) Errors Made by Forensic Labs

Edward G. Brown, Ph.D.
Brown’s Chemistry Services
ed.brown@earthlink.net
1) Methods of Methamphetamine Synthesis
Methamphetamine Manufacturing:

• Two Primary Routes for Synthesis of Meth Used by Clandestine Laboratories in the USA:
  
  – A) Pseudoephedrine Route
    • Precursor is Sudafed from the drug store and involves the use of reducing agents like hydrogen or hydrogen iodide and red phosphorus.
  
  – B) Phenyl Acetone Route
    • Precursors are phenyl acetone or P2P, methyl amine and reducing agents.
Methamphetamine Manufacturing:

Pseudoephedrine Route:

• Substances that can be found at a laboratory that used this route:
  -- Pseudoephedrine or ephedrine;
  -- Hydrogen iodide and red phosphorus; or
  -- A special metal catalyst like palladium, platinum or rhodium and a source of hydrogen gas;
  -- Hydrochloric acid;
  -- Organic solvents like ether, benzene, chloroform, kerosene or white gas; and
  -- Lye or washing soda.
Methamphetamine Manufacturing:

**Pseudoephedrine Route:**

\[
\text{Pseudoephedrine Hydrochloride Salt} \xrightarrow{\text{Reduction of Alcohol Group}} \text{Methamphetamine Hydrochloride Salt and other salts & impurities} \xrightarrow{\text{pH-Selective Purification}} \text{Methamphetamine Freebase} \xrightarrow{\text{Hydrogen Chloride} (= \text{HCl})} \text{Methamphetamine Hydrochloride Salt}
\]
Methamphetamine Manufacturing:

Phenyl Acetone Route:

• Substances that can be found at a laboratory that used this route:
  -- Phenyl acetone;
  -- Methyl amine or methyl amine hydrochloride;
  -- Formaldehyde, ammonia;
  -- Hydrochloric acid;
  -- Aluminum foil, mercuric chloride;
  -- Organic solvents like ether, benzene, chloroform, kerosene or white gas; and
  -- Lye or washing soda.
Methamphetamine Manufacturing:

Phenyl Acetone Route (2):

• In addition, if the phenyl acetone precursor needs to be synthesized first, then other substances that can be found at a laboratory may include:

  -- Magnesium turnings;
  -- Benzyl chloride or benzyl bromide;
  -- Sodium cyanide or acetic anhydride;
  -- Ether or tetrahydrofuran; or
  -- Phenylacetic acid and thorium oxide.
Methamphetamine Manufacturing:

Phenyl Acetone Route:

Phenyl Acetone + Methyl Amine → Phenyl Acetone Methyl Imine

Remove Water → Phenyl Acetone Methyl Imine

Reduction of Imine to Amine

Methamphetamine Freebase

Hydrogen Chloride

Methamphetamine Hydrochloride Salt
Similarities and Differences Between the Two Methods of Manufacture:

• The Phenyl Acetone/ Methyl Amine route requires a source of phenyl acetone (which may need to be synthesized from other starting materials or purchased from another source), methyl amine and a reducing agent such as aluminum amalgam for the first step.

• This reduction procedure produces a product that is a 50: 50 mixture of two isomers (the d-, and l- isomers) of methamphetamine.
Similarities and Differences Between the Two Methods of Manufacture:

• The ephedrine/pseudoephedrine route requires a source of the ephedrine or pseudoephedrine (usually from the drug store) and a reducing agent (like red phosphorus and hydrogen iodide; or hydrogen gas and a metal catalyst).

• This reduction starts with a material that is made up of one naturally-occurring isomer and produces a material that is also one isomer (the d-isomer only) rather than a 50:50 mixture of d- and l-isomers, in contrast to the P2P process.
Similarities and Differences Between the Two Methods of Manufacture:

- For both reaction sequences, once the crude methamphetamine product is made, the purification processes are similar and require various acids (like hydrochloric acid or sulfuric acid), bases (like lye or washing soda) and solvents (like toluene, kerosene or ether) to separate the methamphetamine product from other reaction products.
Similarities and Differences Between the Two Methods of Manufacture:

• Both processes usually have a final step that uses gaseous hydrogen chloride to produce the hydrochloride salt of methamphetamine as a white solid from a solvent solution of methamphetamine free base.

• The waste products from the two methamphetamine synthesis processes differ in the exact chemical make up of the wastes, but there are some similarities, too.
Similarities and Differences Between the Waste Products of Manufacture:

• Typically, meth can be manufactured using flasks and other scientific equipment or it can be produced by simply using quart jars, gallon jugs and coffee filters.

• After a procedure has been used to make meth, what remains (in addition to the final meth hydrochloride product) are solutions of chemical waste. One container has a solution of highly acidic water (pH 1 or less), another has a solution of highly basic water (pH 14 or more), and a third has waste solvent in it. There are also usually one or more filter papers used and left over. Not all filter papers are contaminated with methamphetamine and should be analyzed separately.
Meth Manufacture Summary:

• There are two main methods used to manufacture meth, and each method has variations on the procedures used.

• The primary product from both these variations is methamphetamine hydrochloride, not the freebase.

• The P2P/Methyl amine method produces a product that has two isomers (d- and l- isomers) present in equal amounts. The Sudafed route produces a product that has only one isomer present (the d- isomer).

• Both routes produce chemical wastes. Some of these waste materials are contaminated with meth and others are not.
2) Understanding Lab Reports and Chemical Evidence Lists
Understanding Lab Reports and Chemical Evidence Lists

• Usually, when the discovery evidence is presented to a defense attorney, it consists of:

• A Summary Chemical Analysis Report
  – This is a document from a chemistry laboratory that states if controlled substances were detected or not in various evidence samples.

• A Police Property Report
  – This document lists evidence samples confiscated, weights of materials found, locations where chemical evidence was discovered, etc.
  – Also lists opinion of officer about what the pieces of evidence consist of.
Understanding Lab Reports and Chemical Evidence Lists

• Although the analysis report and police property report are somewhat useful to a defense expert, the most useful documents are usually not included in the initial discovery package; these include:

• A Copy of the Lab Analyst’s Laboratory Notebook:
  – This is a hand-written document from a chemistry laboratory that shows what an analyst did to test the evidence samples.

• A Copy of Each GCMS-, LCMS-, FTIR-Printout:
  – These documents show the machine printouts from the analytical analyses. They can be analyzed to see if the correct procedures were followed or not.
Understanding Lab Reports and Chemical Evidence Lists

• Copies of Other Chemical Evidence Tests that were Run by the Forensic Analyst:
  – These are sometimes hand-written documents and at other times they are typed up summaries. They show what other tests that an analyst did to test the evidence samples.

• A Copy of the Laboratory’s Standard Operating Procedures:
  – These documents show the procedures that the analysts must follow in order to correctly and unambiguously show that controlled substances were detected in the analyst’s experiments.
Understanding Lab Reports and Chemical Evidence Lists
Understanding Lab Reports and Chemical Evidence Lists

\[ \text{Diagram 1: m/z} \]

\[ \text{Diagram 2: m/z} \]
3) Errors and Incorrect Assumptions Made by Forensic Labs in Chemical Evidence Evaluation:
Analytical Testing: Background

Analytical Tools & Methods Commonly Employed in Drug Cases:

• Presumptive Tests:
  - In medical and forensic science, a presumptive test is an analysis of a sample which establishes either:
    - The sample is definitely not a certain substance.
    - The sample probably is the assumed substance.
  - Presumptive tests include: color tests, microcrystalline tests, ultraviolet spectroscopy.
Analytical Testing: Background

Analytical Tools & Methods Commonly Employed in Drug Cases:

• Confirmatory Tests:
  - Tests required to confirm the analysis.
  - Confirmatory tests cost more than simpler presumptive tests, which is why presumptive tests are often made to see if confirmatory tests are necessary.
  - Confirmatory tests include GCMS analyses, LCMS analyses, Infrared Spectroscopy analyses.
Assumption Errors Made by Forensic Labs:

- The forensic analyst relies on the labels of bottles and jars to “prove” the identity of the chemicals inside;
- This is a flawed assumption;
- Labels do not prove the identity of substances inside the containers;
- An expert witness can give an opinion to show that point in court.
Assumption Errors Made by Forensic Labs:

• An analyst performs a presumptive test on your client’s evidence sample but does not perform a confirmatory GCMS test on the evidence.
• This analyst assumes that the presumptive test evidence proves the presence of a scheduled drug.
• This is an incorrect assumption on the part of the analyst.
• Presumptive tests are only a first step; they hint that there may be a certain substance present; it does not prove that the evidence shows the presence of a substance.
• One or more confirmatory tests are required to show that the actual substance is present in the sample.
Assumption Errors Made by Forensic Labs:

• A forensic analyst assumes that a number of samples of white powder pertaining to your client’s evidence all contain a scheduled substance;
• Rather than running separate analyses on the individual samples, the analyst combines all these samples and runs just one confirmatory analysis on the combined sample.
• When a scheduled substance is found in this combined sample, the analyst assumes that each sample contained the substance and attributes the entire weight of the combined samples to your client.
• This assumption is flawed; the substance has not been proven to be in each of the samples separately, so the total weight of the combined samples cannot be used to give a weight for the penalty phase of the case.
GCMS Errors Made by Forensic Labs:

• A blank sample is not run immediately before your client’s sample;
• Cross-contamination by a previous sample run from an unrelated case is possible;
• The analyst has not “proven” that the peak from evidence in the GCMS from your client’s sample is due exclusively to your client’s sample;
• Contamination from a dirty injector needle in the machine from may have occurred;
• The meth product that was detected in your client’s sample may actually be from another evidence sample from a previously-injected evidence sample.
GCMS Errors Made by Forensic Labs:

• A validated sample of the controlled substance is not analyzed immediately after your client’s sample on the GCMS instrument;

• Retention times and MS fragmentation patterns of compounds can vary slightly on a GCMS instrument over time, your client’s sample may be an isomer or closely-related material to the controlled substance without being the actual controlled substance itself;

• A chemistry expert can testify to this point in court.
GCMS Errors Made by Forensic Labs:

• Under certain conditions, pseudoephedrine and ephedrine can be decomposed on the hot injector port of a GCMS instrument to form a small amount of methamphetamine. In these cases, the methamphetamine is being created by the analysis technique, even though it is not present in the sample that is being analyzed.

• Sometimes a forensic analyst makes the incorrect assumption that there is a small amount of methamphetamine present in the analyzed sample.

• A chemistry expert is able to determine if this has occurred in your client’s case or not.
GCMS Errors Made by Forensic Labs:

- Under certain conditions, pseudoephedrine and ephedrine can be decomposed on the hot injector port of a GCMS instrument to form a small amount of methamphetamine. In these cases, the methamphetamine is being created by the analysis technique, even though it is not present in the sample that is being analyzed. This is a false-positive.

- Sometimes a forensic analyst makes the incorrect assumption that there is a small amount of methamphetamine present in the analyzed sample.

- A chemistry expert is able to determine if this has occurred in your client’s case or not.
MS Printout of False Positive

TIC of DATA: 00084A.D

EPOXORINE

MIRROR RESIDUE

SAMPLE 1

"METH"

ACETONE?
MS Printout of False Positive


Abundance vs. Mass/Charge graph with peaks at 42, 58, 63, 77, 105, 117, 132, 146.
Summary of Analytical Section:

• Chemists use a variety of tests to analyze evidence samples.
• Some are presumptive tests and some are confirmatory tests.
• Presumptive tests are only meant to show possibilities; they are not proof that a certain substance is present. They can produce false-positives.
• Confirmatory tests are required to prove that a substance is present. But even then, there can be false-positives or false conclusions drawn.
• There are a number of ways that forensic labs can make mistakes when analyzing chemical evidence.
• An expert for the defense can come to different conclusions than the prosecution experts when analyzing the same evidence samples because the defense expert can see where errors have been made or are possible.
Thank you!