Practical Applications and Examples

9

Every seminar or workshop instructor presents scenarios and practical applications of the information presented. Usually it is a compilation of events in which the presenter applied the techniques under discussion. In essence, the discussion is a series of the presenter's "war stories." In keeping with that tradition, this is the book's "war stories" section. Contained in this chapter is a compilation of scenarios that are used to demonstrate the various points that were brought up in the previous chapters. All of the events actually happened. However, some of the specific details were changed to emphasize the points being discussed.

Reading through the examples may elicit a variety of responses and questions. Some examples may elicit a response like: "That was stupid. Why did he do it that way?" Another instance may elicit an "I did not realize that would happen" response. Hopefully, the absurdity involved in some of the situations will be demonstrated, as they are reviewed in an objective manner. More importantly, the ramifications of the action or inaction on the part of the forensic investigator in the case at hand will be understood.

Other portions of this chapter are technically oriented. The applications will provide insight into how certain examinations are performed and the results of different analytical approaches will be compared and contrasted. In some instances, a step-by-step description of a process will be provided to detail the reasoning behind each of the steps. Understanding the reasoning behind a process assists in incorporating it into specific situations.

Practical Example 1: Extraction Labs

Extraction labs are set up to remove raw materials from a mixture. This is accomplished by using the desired component's physical and chemical prop-

Figure 9.1 Empty over-the-counter cold medication containers.

erties to separate it from the mixture. No chemical change in the raw material occurs during the process. The process, in itself, may not be illegal. However, being able to recognize when the process is being used for illicit purposes provides the expert the ability to fill in missing puzzle pieces.

Clandestine lab operators commonly use over-the-counter medications containing the precursor chemicals needed for the production of amphetamine or methamphetamine. They grind them into a powder, and placed them into a jar. A solvent is added to the powder, and the chemicals of interest are dissolved into the liquid, "extracting" them from the insoluble inert components of the tablet. The liquid is decanted into a glass pie pan that is placed on an electric hot plate to evaporate the solvent. The residue contains a relatively pure form of the desired precursor chemical.

Figures 9.1 and 9.2 show examples of an extraction lab at which three extraction processes are taking place. First, the precursor was chemically removed from the medication with the solvent. Second, the liquid was physically separated from the solids. Finally, the solvent was physically removed from the precursor through evaporation.

Possession of over-the-counter medications is not illegal. The extraction of the precursor chemical components of the tablets may not be illegal. However, the combination of the quantity of tablets and the extraction process may be used to establish intent to conduct an illegal activity.

Practical Example 2: Extraction Labs

Numerous vials of a veterinarian drug preparation containing ketamine were found at a clandestine lab located in a bungalow in a luxury resort. The operators were removing the solution containing the drug from the injection

Figure 9.2 Precursor chemical extraction laboratory.

vials and evaporating the liquid. The resulting powder contained ketamine hydrochloride, which was a controlled substance under local statutes. The operators were convicted of manufacturing a controlled substance in addition to possessing a controlled substance. They "extracted" the drug from the original mixture, which was included in the definition of manufacturing under the local statute.

Practical Example 3: Conversion Labs

Conversion labs are one of the most commonly encountered clandestine labs. In a conversion lab, a raw material is changed into the desired product. This process involves making minor structural changes within the molecule of the compound or of the chemical's salt form. Functional groups may be added or removed from the molecule, somewhat like building with Tinkertoy® pieces. The drug of interest can also be converted from its salt form to the freebase form or from the freebase form to the salt form.

Simply changing the pH of a water solution containing cocaine hydrochloride produces "Crack" cocaine. Changing the pH with a basic reagent chemical removes the hydrochloride component from the cocaine molecule. This changes the cocaine's salt form, making it insoluble in water. It also lowers its boiling point, allowing it to be smoked.

This type of conversion lab can be encountered in a variety of situations. An individual can carry all of the components in his pocket. A vial of water and a small amount of baking soda are all that is necessary to convert cocaine hydrochloride into Crack. Larger-scale operations are slightly less mobile, but the same ingredients are utilized and can be encountered at or in the vicinity of the distribution point.

Figure 9.3 Phencyclidine precursor and reagent chemical.

Practical Example 4: Conversion Labs

The Grignard reagent is a very reactive compound that is commonly used in chemical synthesis. The reaction between bromobenzene and magnesium produces phenylmagnesium bromide, a very reactive Grignard reagent. This compound is an essential intermediate component used in the synthesis of phencyclidine and its analogs. This reaction is self-driven and can be accomplished in a plastic bucket.

In response to information provided by a local street person, investigators found chemicals concealed behind trash cans in an alley (Figure 9.3). A plastic bucket containing a liquid with a strong ether odor with gray metallic particles at the bottom was found a short distance away (Figure 9.4). Laboratory analysis determined that the liquid contained bromobenzene, and the metallic particles were identified as magnesium turnings. The expert concluded that the Grignard reagent located in the plastic bucket was to be added to the phenylcyclohexylcarbonitrile (PCC) that was located in the vicinity to synthesize phencyclidine (PCP).

Practical Example 5: Conversion Labs

Ephedrine or pseudoephedrine is converted into methamphetamine using a simple reduction reaction. In the chemical reaction, a hydrogen atom is substituted for a hydroxyl group (-OH) to produce methamphetamine. The

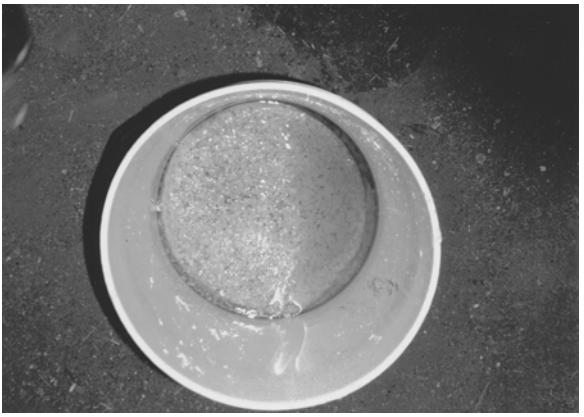

Figure 9.4 Phencyclidine reaction mixture.

skeleton of the molecule is intact. However, the physiological effect of the drug on the body is dramatically different.

Traditionally, the conversion of ephedrine into methamphetamine was accomplished by using a hydrogenator or a reflux apparatus. However, the ingenuity of the clandestine lab operator created a situation in which this conversion can be accomplished using ordinary kitchen utensils. Understanding the basic principles involved in the conversion of the precursor chemical into the final product will allow the investigator to recognize ordinary items that have been adapted for use in a clandestine manufacturing operation.

A clandestine lab chemist was asked if a pressure cooker could be used to manufacture methamphetamine using the ephedrine/HI reduction process. Using the "cooking soup" analogy, the chemist advised the investigator that the process described by the informant was viable and would produce the desired result. A pressure cooker and mason jars were found during the subsequent search of the location. The operator used the mason jars as reaction vessel and condenser. The pressure cooker was used as the heating mantle. A condenser was not required, because the closed mason jars inside the pressure cooker produced a closed system that contained the fumes that would normally have been condensed or vented away from the reaction. Figures 9.5 and 9.6 show examples of a pressure cooker that was used to produce methamphetamine in this fashion.

Practical Example 6: Synthesis and Extraction

The synthesis process is a chemical reaction or series of chemical reactions in which molecules or parts of molecules are combined to create a new

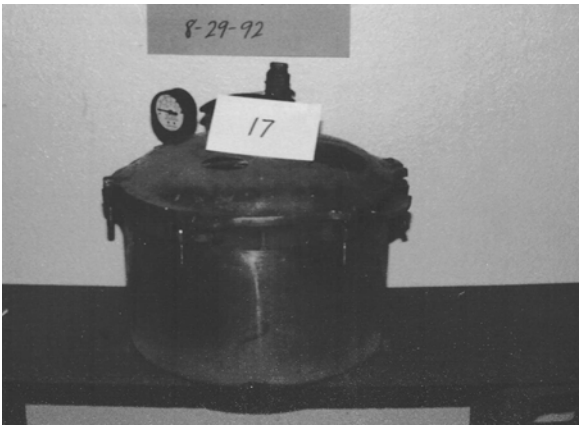

Figure 9.5 Pressure cooker reaction vessel.

Figure 9.6 Interior of pressure cooker.

molecule. This process can be equated to a chemical-type Erector® set. It differs from the conversion process in that the skeleton of the resulting molecule is a sum of the molecules or significant parts of the molecules involved in the reaction. Lysergic acid diethylamide (LSD), phencyclidine (phenylcyclohexyl piperidine, PCP), phenylacetone (P2P), and certain methamphetamine reactions are examples of drugs produced using the synthesis process.

Phencyclidine is produced in a multistep reaction. During the process, bromobenzene, cyclohexanone, and piperidine are chemically combined in a two-step process. The resulting molecule has the combined chemical skeletons of all three precursor chemicals.

Figure 9.7 Bucket chemistry example of PCP lab.

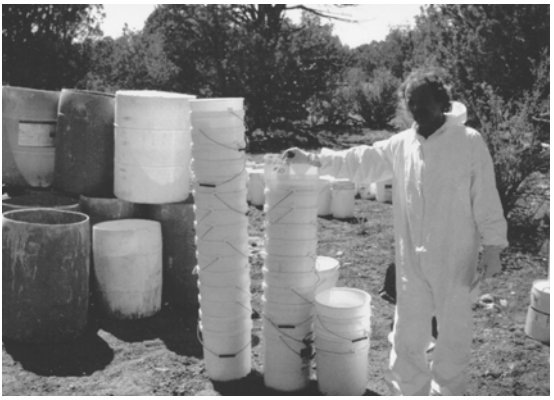

Figure 9.8 Large-scale PCP lab.

The manufacturing of PCP is so simple that it is commonly described as "bucket chemistry." The equipment required for this synthesis reaction is shown in Figures 9.7 and 9.8. The images demonstrate that the required equipment can be common and ordinary and does not have to be sophisticated or exotic. In this example, it was estimated that over 100 pounds of PCP was produced in a rural operation, in which all of the chemical reactions were conducted in 5 gal plastic paint buckets.

Practical Example 7: Distillation

A distillation apparatus can be used to simultaneously synthesize and extract phenylacetone. The precursor and reagent chemicals are combined in the

reaction flask and heated to a rolling boil. As the mixture boils, the chemicals react, producing phenylacetone and its associated reaction by-products. The by-products, with a boiling point lower than phenylacetone evaporate, are separated, collected, and discarded through the distillation process. Once the boiling point of the reaction mixture reaches that of the phenylacetone, the separated liquid is saved. The heat is removed from the reaction when the mixture's temperature rises above the boiling point of phenylacetone.

Practical Example 8: Distillation

Figure 9.9 illustrates an example of a situation in which a little knowledge can be dangerous. The operator in this situation understood the basic concept concerning distillation. However, he arranged a distillation apparatus such that the reception flask was above the boiling flask. This arrangement resulted in nothing more than a modified reflux apparatus. Gravity returned the liquid from the condensing vapors to the boiling flask instead of separating it into the reception flask. The operator had no idea why the apparatus was not working.

Practical Example 9: Distillation

The operator in the situation illustrated in Figure 9.10 constructed a vacuum distillation apparatus to purify phenylacetone. The boiling container was constructed from an 8" steel pipe with metal plates bolted onto the top and bottom. A kitchen hot plate was used to apply heat to the boiling container. The condenser was constructed of two sizes of copper tubing. Cool water was circulated through the makeshift condenser using a submersible water pump and a trash can containing ice water. The reception flask was a vacuum flask connected to a beer keg that was connected to a commercial vacuum pump. This apparatus functioned very well.

Practical Example 10: Extraction and Separation

During the final stages of a methamphetamine synthesis or conversion reaction, the reaction mixture is cooled to room temperature. The pH of the solution is changed, and an organic solvent is added to the mixture. The methamphetamine dissolves in the organic layer. The liquid combination is placed into a separate container, and the organic layer is isolated and separated through the use of a traditional separatory funnel (Figure 9.11). The liquid combination is placed into the funnel and allowed to form two distinct

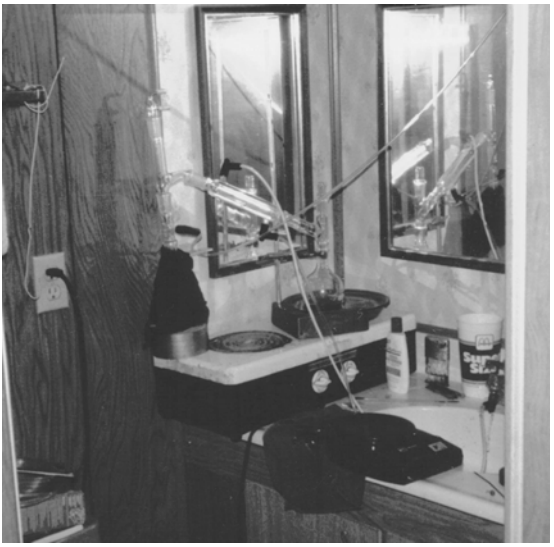

Figure 9.9 Inverted condensing column on a distillation apparatus.

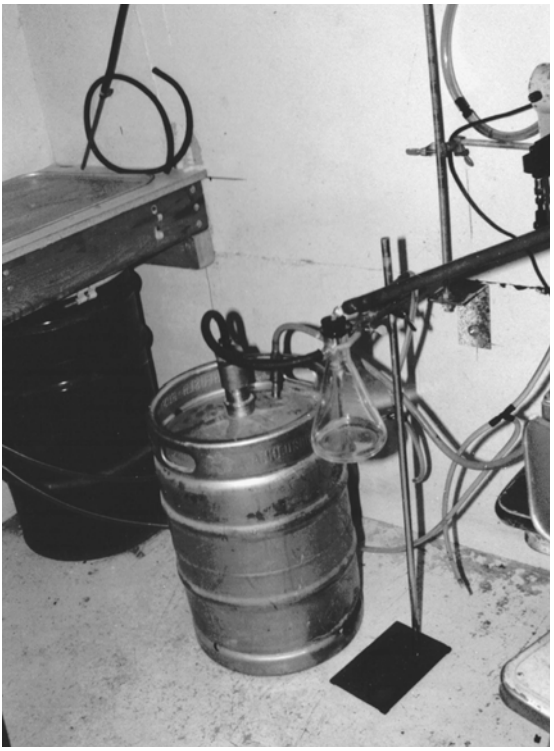

Figure 9.10 Homemade vacuum distillation apparatus.

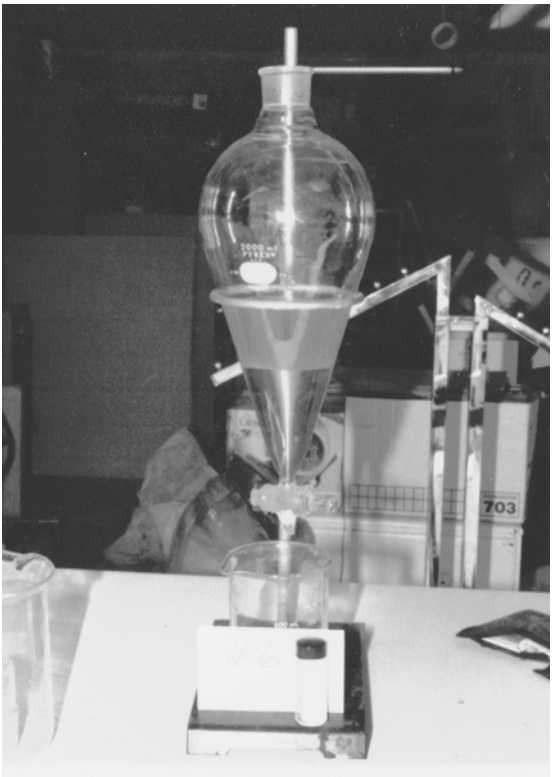

Figure 9.11 Conventional separatory funnel containing a two-phase liquid.

layers. The valve is opened, and the lower liquid is allowed to drain into a receptacle. The valve is then closed when the line defining the separation between the top and bottom layers reaches the valve.

Sport bottles (Figure 9.12) can be used in a similar manner. The combination of liquids is placed into the bottle, the cap is put into place, the liquids are allowed to separate, and the bottle is then inverted. A vacuum is created in the air space above the liquids, keeping the liquids from pouring out of the bottle. The lower liquid is removed by gently squeezing the bottle, forcing the liquid out of the restricting valve at the opening.

Practical Example 11: Filtration

Hydrogen chloride gas is added to the organic solution from Practical Example 10. The freebase drug and the gas react to produce the hydrochloride salt form of the drug, which is insoluble in the organic liquid. The solid–liquid mixture is poured into a Buchner funnel attached to a vacuum flask (Figures

Figure 9.12 Plastic soda bottle used as a separatory funnel.

9.13 and 9.14). The liquid is drawn into the vacuum flask when the vacuum is applied to the system, leaving the solid in the Buchner funnel. Acetone can be used to remove the reaction by-products from the solid. It is poured over the solid and drawn by vacuum into the flask. As the by-products are removed, the solid turns white.

In this example, the drug was chemically extracted from the liquid by changing its salt from. The solid was then physically extracted from the liquid through vacuum filtration. Finally, the reaction by-products were chemically and physically extracted from the drug in a tandem operation. The acetone chemically extracted the by-products, and was simultaneously physically extracted from the drug, while being filtering under vacuum.

Practical Example 12: Mechanical Explosions

A mechanical explosion occurs when the structural integrity of a container is compromised as the result of excess pressure inside the container. This

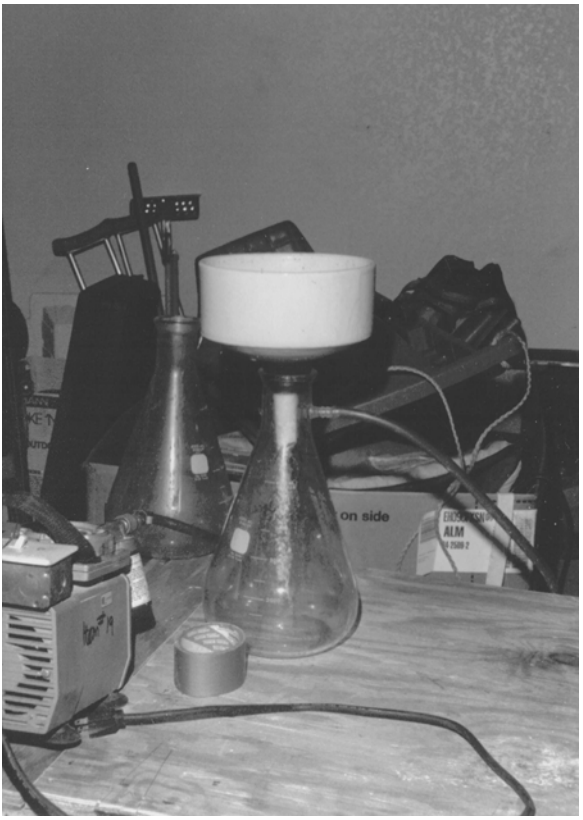

Figure 9.13 Vacuum filtration.

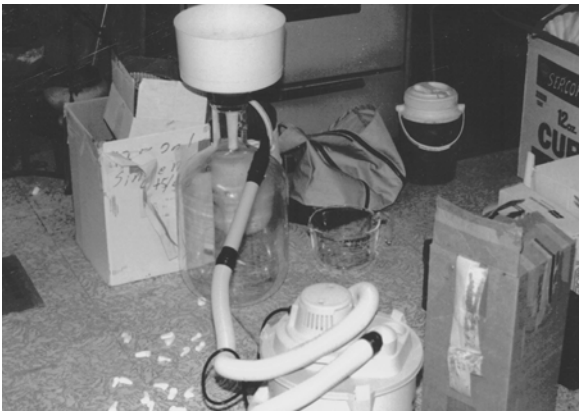

Figure 9.14 Vacuum filtration with shop vac as vacuum source.

situation can occur by design, as in the case of a pipe bomb. However, when associated with a clandestine lab, it is more often a result of equipment modification performed by the operator. The following examples are results of operator equipment modifications.

The operator attempted to vent the fumes emanating from the top of a reflux condenser into a makeshift filtering device. As a result, the opening of the condensing column became obstructed. The strength of the boiling flask was compromised by stress cracks that were the result of improper handling. The pressure would build to a point at which one of three things could happen. First, the pressure could clear the obstruction at the top of the condensing column. The excess pressure would be vented into the filtering device. This may or may not have an adverse effect, depending upon the construction of the filter.

Second, the connection between the boiling flask and the condensing column could release. The pressure in the system would then propel the condenser like a missile into the ceiling. The contents of the reaction flask would spew from the reaction flask opening like a fountain, bathing everything in the area with boiling hazardous chemicals.

The other option is that the reaction flask could lose its structural integrity. It would shatter, and the pressure from the system would propel the broken pieces of glass like shrapnel from a grenade and coat the immediate area with the boiling reaction mixture. This option contains the dual hazard of chemical exposure and of being impaled by reaction flask fragments.

Practical Example 13: Mechanical Explosions

A clandestine lab operator repeatedly used a kitchen pressure cooker as a reaction vessel. Over time, the pressure relief valve corroded, so he soldered the opening closed. This particular operator placed the reaction mixture directly into the altered pressure cooker, placed it on the electric kitchen stove, and turned on the stove. The acids in the reaction mixture weakened the metal during repeated use of the pressure cooker. The pressure from the boiling reaction mixture reached the point at which the pressure cooker lost its structural integrity and exploded. The metal lips on the pot portion, which held the lid on, sheered off. The pressure propelled the lid into the stove vent, spewing hot reaction mixture over the immediate area. Fortunately, no one was in the vicinity of the kitchen at the time of the explosion.

Practical Example 14: Vapor Explosions

The operator of a large-scale gamma hydroxy butaric acid (GHB) operation constructed a drying room in which to evaporate the residual acetone from

Figure 9.15 Vapor explosion fire damage.

Figure 9.16 Vapor explosion blast damage.

his final product. During the drying process, the concentration of the acetone vapors inside the drying room reached the explosive range. A spark was generated inside the drying room when the operator turned on an interior light. This resulted in a vapor explosion with effects that are demonstrated in Figures 9.15 and 9.16.

Practical Example 15: Compressed Gas Hazards

Compressed gases are utilized in a variety of situations in the clandestine manufacture of controlled substances, and they pose multiple hazards. First,

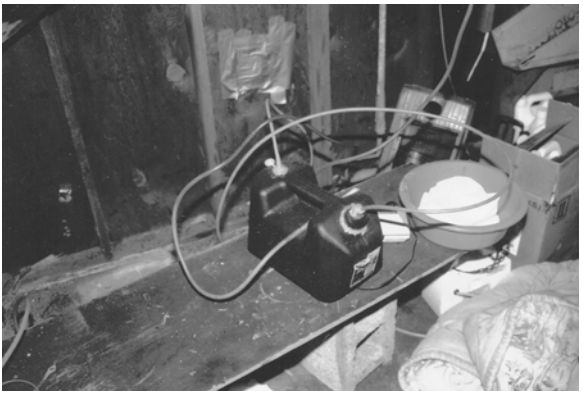

Figure 9.17 Hydrogen chloride gas generator.

the chemical within the container may have a hazardous component. Second, the container may be unstable and pose a physical hazard to anyone attempting to handle it. Third, because of the second condition, there may be no safe way to determine the status of the first. Simply put, the investigator does not know what is in the container, and the container's condition may be too hazardous to determine what is inside. The following examples are used to provide some insight as to the hazardous potential of compressed gas containers encountered in clandestine labs.

Hydrogen chloride gas ($HCl_{(g)}$) is used to convert freebase drugs into the hydrochloride salt form. One method bubbles commercially available $HCl_{(g)}$ into a mixture of extraction solvent and freebase drug. The freebase drug reacts with the $HCl_{(g)}$, creating a solid that precipitates out of solution. Clandestine lab operators commonly generate their own $HCl_{(g)}$ using household chemicals. They place the chemical mixture into containers, such as compressed gas cylinders, plastic gas containers, or other containers that can be sealed and in which the pressurized gas is vented in some manner. The result is a pressurized container of $HCl_{(g)}$.

The metal that compressed gas cylinders are constructed of is incompatible with the $HCl_{(g)}$. The $HCl_{(g)}$ corrodes the brass valves or reacts with the metal of the container. The corroded valve can break during use, or the container may eventually lose its structural integrity. Both situations lead to a discharge of pressurized $HCl_{(g)}$.

The other situations are not much safer. The containers used by the operators may be resistant to the corrosive nature of the chemicals involved, but they are not designed to withstand significant pressures. As a result, $HCl_{(g)}$ is continually placed into the atmosphere until the chemical reaction between the ingredients has finished.

Practical Example 16: Compressed Gas Hazards

Clandestine lab operators utilizing the Birch reduction (commonly referred to as the "Nazi" method) obtain the liquid ammonia required from agricultural areas that use it as a fertilizer. The operators use the propane tank from a gas barbecue to transport and store the stolen ammonia. Over time, the ammonia corrodes the valve on the tank to the point at which the valve does not function. It may break when operated, resulting in the release of pressurized ammonia into the atmosphere.

Practical Example 17: Compressed Gas Hazards

Under pressure, ephedrine can be reduced into methamphetamine in the presence of a catalyst, acid, and hydrogen. Clandestine lab operators have designed an apparatus that uses a 2 l plastic soda bottle that will facilitate the hydrogenation process. Under normal conditions, plastic soda bottles can maintain pressures in excess of 500 lb/in^2. However, the conditions in a hydrogenation reaction expose the bottle to temperatures and chemicals the container was not designed for. Constant increases and decreases of pressure during the hydrogenation process, coupled with the heat generated by the chemical reaction, compromise the structural integrity of the bottle to the point where any shock will cause the plastic skin of the bottle to rupture and peel open like an over-ripe watermelon, releasing pressurized hydrogen and corrosive chemicals.

Practical Example 18: Initial Crime Scene Evaluation

Everything done at a crime scene potentially has evidentiary value. Every action and word has the potential of finding its way into the court. Nowhere was this more apparent than in the O. J. Simpson case, in which the investigators spent hours on the witness stand explaining comments and personal opinions that were expressed during the crime scene investigation. Provided in this section are examples of how actions taken at a clandestine lab scene may have ramifications in later stages of the investigation.

A clandestine lab scene chemist and the lead investigator began processing the scene of a suspected clandestine drug lab after the scene had been secured and the hazards were abated. The lead investigator, who only had a minimal knowledge of clandestine manufacturing techniques, immediately began videotaping the scene and providing audio descriptions of how each chemical and piece of equipment would be used to manufacture methamphetamine.

The scene chemist, who had 10 years of experience in clandestine lab processing, was performing a preliminary walk through at the same time. Within minutes, the scene chemist realized that the operator was manufacturing explosives, not drugs. He stopped the processing and evacuated the lab area to revise the processing plan. When reading the physical evidence, the scene chemist realized the operator was manufacturing nitroglycerine, not methamphetamine. This piece of knowledge radically affected how the balance of the scene was processed.

Practical Example 19: Training and Experience

A forensic chemist who was not trained in clandestine lab manufacturing methods arrived at the scene of a suspected methamphetamine lab. Without evaluating the combination of chemicals and equipment that was present at the scene, he pronounced that the operation was manufacturing methamphetamine. He took a minimal amount of samples and left the scene. Using the scene chemist's opinion, without corroborating laboratory analysis, the lead investigator charged the operator with manufacturing methamphetamine.

A clandestine lab chemist subsequently evaluated the physical evidence from the scene. His laboratory examinations of the evidentiary samples, evaluation of the scene's photographs, and review of the chemicals revealed that the operator was manufacturing diethyltriptamine, a hallucinogen, not methamphetamine. The haste actions of the scene chemist and the lead investigator led to the dismissal of the charges against the operator.

Practical Example 20: Training and Experience

In contrast to Practical Example 19, demonstrated in this practical example are the positive effects of having qualified personnel read the physical evidence and adjust the scene processing protocols as more information is discovered.

A clandestine lab response team properly secured and abated the hazards at a site run by an educated commercial operator who had a history of manufacturing gamma hydroxybutaric acid (GHB). The operation under investigation was clean and nonoperational. During their evaluation of the chemicals and the operator's notes, the scene chemists determined that the operator was experimenting with the manufacture of meperidine and fentanyl analogs. These compounds have been linked to Parkinson's disease. At this point, the hazard potential dramatically changed, as did the approach to the way the scene would be processed. The lab area was evacuated, and the scene-processing plan was revised.

Practical Example 21: Sampling

The following is an example of the ramifications of improperly sampling a clandestine lab scene. The scene chemist in this situation did not possess training in clandestine lab scene processing. He did an admirable job of photographing the scene, which allowed the clandestine lab chemist to render some opinions after the fact.

Three containers located at the scene of this clandestine drug lab were identified as containing an acid. One container was a plastic gasoline container containing a clear acidic liquid that produced white fumes when the container was opened. The two other containers were commercial 500 ml clear glass bottles with black caps. The labels on the bottles had been removed. The bottles contained a clear acid liquid with a yellow tint. Photographs were taken of each of the containers. Only the red plastic container was sampled. Subsequent laboratory analysis of the sample revealed that the contents were consistent with hydrochloric acid.

Below is an excerpt of the analytical chemist's testimony. The defense contended that lack of the presence of hydriodic acid precluded the operator from manufacturing the controlled substance that the state contended. Cross-examination of the analytical chemist charged with analysis of the evidence proceeded as follows:

Defense Attorney: You stated exhibit 12 contained hydrochloric acid?
Chemist: Yes sir.
Defense Attorney: So there was no hydriodic acid found at the scene?
Chemist: No Sir, I cannot say that.
Defense Attorney: But your report states that you found hydrochloric acid, not HI. How can you say that there was hydriodic acid present?
Chemist: The items in exhibit 24 and exhibit 31 were not sampled. The packaging and the color of the liquid are consistent with hydriodic acid.
Defense Attorney: But your report states that hydrochloric acid was the only acid identified.
Chemist: That is correct. However, the items in exhibit 24 and 31 were not sampled so I could not analyze the contents. Without laboratory analysis I cannot comment on the contents.
Defense Attorney: So you are saying you did not find any hydriodic acid?
Chemist: What I am saying is that I cannot say that there was no hydriodic acid at the scene. The packaging and color of the liquid of items 24 and 31 is consistent with commercially packaged hydriodic acid.

This whole exchange could have been avoided if the items at the scene were sampled properly. This would have provided the analytical chemist the opportunity to identify the contents of each container.

Practical Application 1: Bottle Volume Estimates

In some jurisdictions, the penalty associated with crime is related to the amount of controlled substance seized. Many statutes use the phrase, "at time of seizure," as the benchmark time. In simple possession or possession for sale cases, this value is easily determined by weighing the substance on a calibrated balance during the laboratory examination of the exhibit. This can be problematic in clandestine lab investigations, because a majority of the evidence is disposed of due to its hazardous nature. The 2 fluid oz sample that is retained for laboratory examination may hardly be representative of the volume of substance that was seized. Without documentation to support a larger volume/weight, the only value the court can rely on to establish a sentence would be the weight/volume of the representative sample that was submitted for laboratory examination. If the dimensions of the containers and their contents are documented, simple geometry can be used to establish the original volume of the substance. Thus, this will give the court the "at the time of seizure" value to use to establish the sentence.

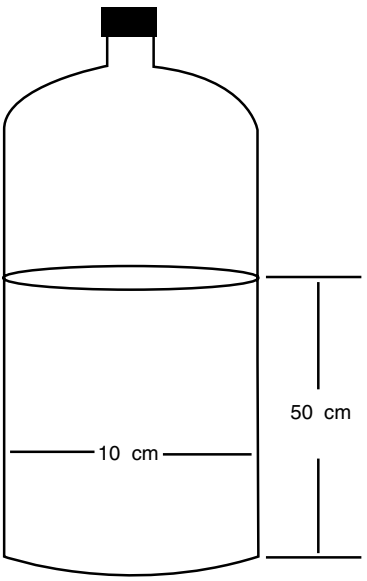

Figure 9.18 Bottle containing liquid.

The volume of the contents of a clear glass bottle must be determined. The cylindrical-shaped bottle has a diameter of 10 cm. The height of the liquid in the bottle is 50 cm. The volume of the liquid can be calculated using the simplified cylinder volume equation (Figure 4.1) as follows:

$$\text{Volume}_{\text{cylinder}} = 0.78 * \text{diameter} * \text{diameter} * \text{height}$$
$$= 0.78 * 10\text{ cm} * 10\text{ cm} * 50\text{ cm} = 3900\text{ ml} = 4.1\text{ qt}$$

Practical Application 2: Flask Volume Estimates

The volume of a clear yellow liquid in an Erlenmyer flask is needed. The Erlenmyer flask has a conical shape, is 30 cm tall, and has a base diameter of 20 cm. The height of the liquid in the flask is 10 cm, and the diameter of the flask at the top of the liquid is 10 cm.

Calculating the volume is a three-step process. The total volume of the flask is calculated first. Second, the volume of the air on top of the liquid is calculated. Finally, the air volume is subtracted from the total volume to determine the volume of the liquid in the flask. Using the equation from Figure 4.2, the calculation is as follows:

$$\text{Volume}_{\text{total}} = 0.26 * \text{diameter} * \text{diameter} * \text{height}$$
$$= 0.26 * 20\text{ cm} * 20\text{ cm} * 30\text{ cm} = 3120\text{ ml}$$

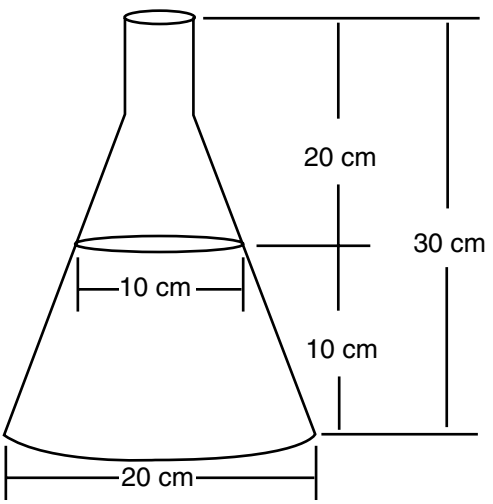

Figure 9.19 Flask containing liquid.

$$\text{Volume}_{\text{air}} = 0.26 * \text{diameter} * \text{diameter} * \text{height}$$
$$= 0.26 * 10\text{ cm} * 10\text{ cm} * 20\text{ cm} = 520\text{ ml}$$

$$\text{Volume}_{\text{bottom}} = \text{Volume}_{\text{total}} - \text{Volume}_{\text{air}}$$
$$= 3120\text{ ml} - 520\text{ cm} = 2600\text{ ml} = 2.7\text{ qt}$$

Practical Application 3: Separatory Funnel Volume Estimates

A separatory funnel is found containing a two-phase liquid. The bottom layer is 5 cm high, and the top layer is 10 cm high. The diameter of the funnel at the point the two liquids meet is 10 cm. The diameter of the funnel at the top of the top liquid is 20 cm. What is the volume of both liquids?

The basic shape of a separatory funnel is that of an upside-down cone. Therefore, the same method that was used to calculate the volumes in the flask is used for this calculation. The total volume in the funnel occupied by liquid is the first thing to be calculated. The next step is to calculate the

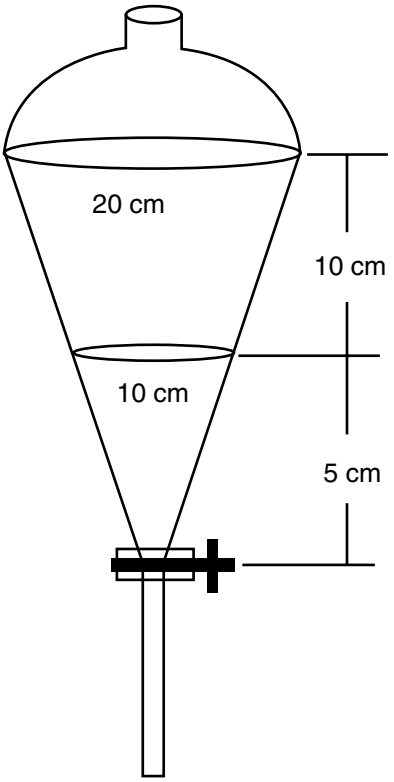

Figure 9.20 Funnel containing liquid.

volume in the liquid phase closest to the apex of the cone. Finally, the lower layer volume is subtracted from the total volume to establish the volume of the upper layer.

$$\text{Volume}_{\text{total}} = 0.26 * \text{diameter} * \text{diameter} * \text{height}$$
$$= 0.26 * 20\text{ cm} * 20\text{ cm} * (10\text{ cm} + 5\text{ cm}) = 1560\text{ ml} = 1.64\text{ qt}$$

$$\text{Volume}_{\text{bottom}} = 0.26 * \text{diameter} * \text{diameter} * \text{height}$$
$$= 0.26 * 10\text{ cm} * 10\text{ cm} * 5\text{ cm} = 130\text{ ml} = 0.5\text{ cup}$$

$$\text{Volume}_{\text{top}} = \text{Volume}_{\text{total}} - \text{Volume}_{\text{bottom}}$$
$$= 1560\text{ ml} - 130\text{ cm} = 1430\text{ ml} = 1.51\text{ qt}$$

Practical Application 4: Reaction Flask Volume Estimates

The calculations to establish the volume of a sphere that is filled with liquid are more complicated than the simple mathematics used in the previous examples. In July, 1991, the DEA published a table of partial sphere volumes based on reaction flask size and liquid height. This table was reproduced in Appendix P. The diameter of the reaction flask is used to establish its volume. The height of the liquid in the flask is then cross-referenced in the table in Appendix P to obtain the amount of liquid in the reaction flask.

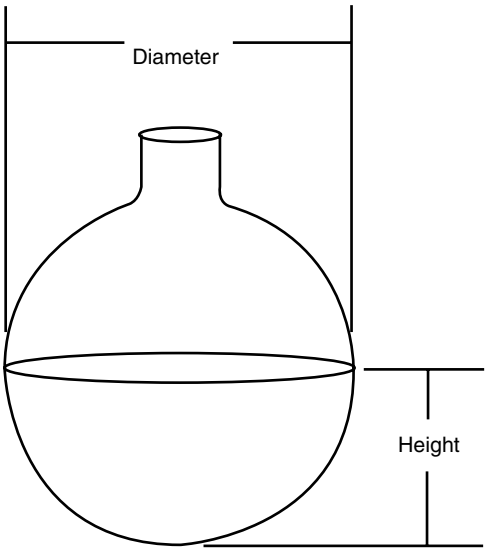

Figure 9.21 Reaction flask containing liquid.

Practical Example 22: Data Interpretation

The laboratory analysis of samples taken at the scene of a clandestine laboratory can generate a significant amount of information. The analytical methods traditionally utilized by the forensic chemist analyzing the samples can provide the answers to the questions the expert needs to provide an informed opinion concerning the operation under investigation. However, the analytical chemist needs to know what questions should be answered so he can apply the appropriate analytical technique to the samples being analyzed. In this section, a few examples of how the information provided by the analytical chemist is used by the clandestine lab chemist to reach his conclusions are provided.

The inorganic analysis of an off-white crystalline substance from a suspected methamphetamine lab reveals the presence of sodium, phosphorous, iodine, and a relatively small amount of chlorine. The chemist must account for the presence of each of the elements. From the notes seized at the scene and the chemical inventory, it is the chemist's opinion that the operator was probably using the HI/red phosphorous method of reducing ephedrine to methamphetamine. Using this as a basis, the chemist reasons that the iodine originated from the HI, the phosphorous was from the red phosphorous, and the sodium hydroxide used to neutralize the HI contributed the sodium. Finally, the small amount of chlorine is assumed to have originated from the hydrogen chloride salt of the ephedrine that was used as a precursor chemical.

Practical Example 23: Data Interpretation

A red sludge material recovered from a trash search was submitted for laboratory analysis to help develop the probable cause necessary for a search warrant for the location of a suspected clandestine lab. The chemist is asked what method was being used to manufacture the methamphetamine without being supplied a list of chemicals or recipe from the operator. To provide the requisite opinions, the clandestine lab expert combines the information from the organic and inorganic chemical profiles to determine the controlled substance being manufactured and the method of manufacturing.

The organic analysis of the sample revealed the presence of methamphetamine, ephedrine, and a trace of phenylacetone. The ratio of these components indicated the operator was reducing ephedrine to methamphetamine using hydriodic acid. This was consistent with the chemist's expectations from his visual inspection of the sample prior to analysis.

The inorganic analysis revealed the presence of potassium, iodine, phosphorous, and chlorine in relatively equal amounts, with a small amount of

sulfur present. This inorganic profile presented a quandary. It was not consistent with the information provided by the organic analysis. The unusually high concentration of chlorine and potassium, in combination with the presence of sulfur, were of concern to the clandestine lab chemist.

To rectify the discrepancy between the organic and inorganic results, the clandestine lab chemist had to think "outside the box," utilize his knowledge of organic synthesis, and incorporate alternative methods used by clandestine lab operators in the past. He referred to a method that substitutes a strong acid and a source iodide, usually from an iodine salt, to reduce the ephedrine. Using this information, he proposed that the potassium and iodine were from a potassium iodide salt, the chlorine was from hydrochloric acid, and the phosphorous was from the red phosphorous. The small amount of sulfur was attributed to the sulfate salt form of the ephedrine that was used as a precursor. When the operation was seized, the chemical inventory and the operator's notes confirmed the chemist's proposal.

Practical Example 24: Data Interpretation

All samples should be given the same value and analytically be treated the same. It is easy for the analytical chemist to become complacent and dismiss a sample, because it "looks" a certain way. There is some validity to the initial observations of a seasoned analytic chemist. However, his "gut feeling" should not be substituted for a documentable scientific analysis. The following example demonstrates how misleading gut feelings can be.

An analytic chemist received a sample of a clear liquid seized from a freezer located in a clandestine lab that was operating in a government laboratory. The sample was from a labeled container, and its odor and general appearance were consistent with the label. The chemist was prepared to dismiss the sample as "contents consistent with the label" without analytical data to support his conclusion. However, much to his surprise, he detected a significant amount of methylenedioxyamphetamine (MDA) when he performed a routine gas chromatographic screen on the sample.

Practical Application 5: Dry Extractions

There are a number of different extraction techniques available to the analytic chemist. Each has its place in the toolbox of the analytical chemist. The challenge is for the analytical chemist to choose the one that is most appropriate for the sample under examination. The following practical examples are applications of various extraction techniques used in the analysis of clandestine lab samples.

Cocaine HCl is generally found in a powder matrix mixed with one or more adulterants or diluents. Many of these compounds can be removed through one or more dry extractions. The following are two techniques that can be used to isolate cocaine HCl without altering its salt form:

Dry Extraction 1
- Physically isolate particles that appear to be pure cocaine HCl with tweezers.
- Analyze the particles using IR, which identifies the cocaine as well as establishes the salt form. The sample can be analyzed via GC/MS if the salt form of the cocaine is not an issue.

Dry Extraction 2
- Dissolve powder sample in chloroform. (Cocaine HCl is soluble in chloroform, and most common diluents and adulterants are not.)
- Separate the chloroform from the powder.
- Evaporate the chloroform.
- Analyze the residue via IR to confirm the presence of cocaine as well as establish the salt form. The sample can be analyzed using GC/MS if the salt form of the cocaine is not an issue.

Dry Extraction 3
- Dry wash the powder sample with ethyl ether. (This removes assorted impurities, i.e., niacinamide and any freebase cocaine that might be present.)
- Dry wash the sample with acetone. (This will remove common diluents, i.e., lidocaine HCl.)
- Analyze the residue via IR to confirm the presence of cocaine as well as establish the salt form. The sample can be analyzed using GC/MS if the salt form of the cocaine is not an issue.

These extractions may or may not remove all of the auxiliary components from the sample, allowing it to be confirmed as cocaine. However, enough of them should be eliminated to establish if a salt form exists and, if so, which one it is.

Practical Application 6: Methamphetamine Extraction

Just as the salt form of cocaine may be significant, the salt form of methamphetamine may affect how the defendant is initially charged or ultimately sentenced after conviction. The salt form may additionally provide information as to the manufacturing method the operator was using. Again, the chemist must refer to the statutes he is working under to determine whether or not a salt determination is necessary.

The differentiation of pure methamphetamine base and a methamphetamine salt, usually HCl, is straightforward. The freebase form of methamphetamine is an oily liquid. The HCl salt form is a crystalline solid. The freebase is soluble in most organic solvents. The HCl salt is a solid and is soluble in common solvents like methanol and chloroform but is insoluble in ether, acetone, Freon, and hexane.

The simplest way to determine the salt form of methamphetamine in a volatile organic solvent is to evaporate the solvent and examine the residue. If the residue is a liquid, chances are that the freebase form is present. If a solid residue remains, some sort of salt form is indicated. In either case, IR analysis would confirm the methamphetamine and identify the salt form, if the sample is not contaminated with reaction by-products.

Practical Application 7: Methamphetamine Extraction

Methamphetamine salts are soluble in acidic aqueous solutions, i.e., reaction mixtures. There are times when the salt form of methamphetamine can be used to establish a manufacturing route. The following extraction can be used to remove methamphetamine HI from a reaction mixture without altering the salt form:

Wash an acidic aqueous liquid, or red reaction sludge with ether to remove many of the neutral organic by-products.
Wash the sample with chloroform to remove the mineral acid salts of methamphetamine that may be soluble in chloroform.
Isolate and evaporate the chloroform.
Analyze the residue for methamphetamine and its associated salt form via IR.

Practical Application 8: Ephedrine/Pseudoephedrine Separation

Ephedrine and pseudoephedrine can be reduced to methamphetamine. Their GC retention times and their mass spectra are essentially the same, under the conditions generally used in forensic drug identification. Without derivatization, they are generally considered indistinguishable. Most other screening tests cannot differentiate the two. Their solubility differences can be used to separate them (pseudoephedrine HCl is soluble in chloroform, ephedrine HCl is not). The following extraction can be used to separate pseudoephedrine HCl from ephedrine HCl:

Dry wash the powdered mixtures of ephedrine HCl and pseudoephedrine HCl with chloroform.
Analyze the dry-washed solid via IR for ephedrine HCl.
Isolate and evaporate the chloroform.
Analyze the residue via IR for pseudoephedrine HCl.

Practical Application 9: Methamphetamine By-Product Profile Extraction

Powdered methamphetamine samples can be a wealth of information, concerning not only the salt form of the methamphetamine but also the presence of reaction by-products and any adulterant or diluents that may be present. The following is a series of extractions that can be used on powdered methamphetamine samples:

Dry wash the sample with acetone to remove niacinamide and reaction by-products.
Isolate the acetone.
Add hexane to the isolated acetone to precipitate out any existing niacinamide.
Isolate and analyze the precipitate by IR.
Analyze the acetone/hexane mixture for the reaction by-products by GCMS or GCFTIR.
Dry wash the solid sample with chloroform to remove the methamphetamine salts.
Isolate and evaporate chloroform.
Dry wash residue with acetone.
Analyze residue by IR to confirm methamphetamine and determine the salt form.
Dry wash the sample with methanol.
Isolate and evaporate the methanol.
Analyze the residue for ephedrine.

Practical Application 10: Quantitation

Determination of the actual amount of controlled substance in a sample is not generally required to establish the facts of a case. However, this knowledge provides investigators and prosecutors with information that can be used to develop investigative leads or to demonstrate what portion of the process the operation was in at the time of seizure. Quantitation information can also

be used as part of a quality control mechanism within the laboratory. The quantitation method used depends on the information desired and the level of accuracy required. In this section, how different quantitation methods can be employed will be described.

In one application, the concern is determining the amount of pure controlled substance at the time of seizure. As a general rule, the analytical chemist only sees a representative sample of an exhibit. To determine the amount of controlled substance that was in the original container, he must determine the concentration of the sample presented to him. He then uses this value and the original volume to calculate the amount of controlled substance in the original container.

Practical Application 11: Gravimetric Quantitation

To obtain the concentration of the sample, the chemist begins with a 10 ml sample. He performs a series of extractions to isolate the previously identified methamphetamine as the hydrochloride salt. He then divides the weight of the resulting residue (0.10 g) by the volume of the sample to obtain the concentration. The concentration value of the sample can then be multiplied by the original volume of liquid of the item seized (1000 ml) to obtain the amount of methamphetamine in the original container. The following is the resulting calculation sequence:

$$\text{Concentration}_{\text{sample}} = \text{Weight}_{\text{extracted methamphetamine}}/\text{Volume}_{\text{sample}}$$
$$= 0.10\ \text{g}/10\ \text{ml} = \mathbf{0.010\ g/ml}$$

$$\text{Weight}_{\text{original container}} = \text{Original Volume} * \text{Concentration}_{\text{sample}}$$
$$= 1000\ \text{ml} * 0.010\ \text{g/ml} = 10.0\ \text{g}_{\text{in original container}}$$

Practical Application 12: Serial Dilution Quantitation

Plotting a graph of the sample concentration versus the instrumental response of a gas chromatograph produces a line that can be used to calculate the concentration of an unknown sample. This calculation technique is demonstrated by using the following scenario.

A series of three solutions with a known concentration of Compound *A* are prepared. Their concentrations are 3 mg/ml, 2 mg/ml, and 1 mg/ml, respectively. A 3 mg/ml solution of the questioned sample, which contained an unknown amount of Compound *A*, was also prepared. The peak areas from the gas chromatograph analysis were 3000, 2000, 1000, and 1275, respectively. The concentration of the unknown mixture can be extrapolated

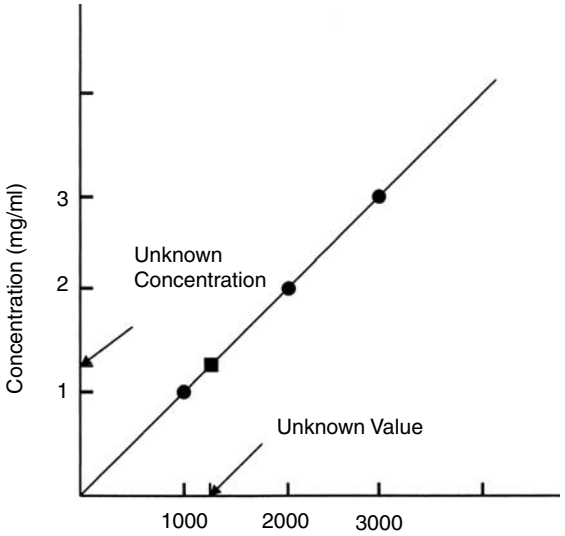

Figure 9.22 Beer's law plot.

directly from the graph. The accuracy and precision of this method is subject to the size of the graph paper and the eye of the chemist (Figure 9.22).

Practical Application 13: Mathematic Application of Serial Dilution Quantitation

The modern gas chromatographs produce precise analytical data that generate a linear response on a graph of concentration versus instrument response. The line tends to travel through the origin, making use of mathematical calculations an option. Therefore, the mathematical equation of a line ($y = mx + b$) can be used to calculate the value of the concentration of the unknown. This removes the subjectivity introduced by the size of the graph paper and the thickness of the pencil that are used to chart the concentration in the previous example.

The first step is to establish the slope of the line (the m value), which is the difference in concentration (the rise) divided by the difference in instrumental response (the run). Using the extreme values of the standard solution, provide a concentration range within which the unknown sample will most likely fall. Using the data from the previous application, the slope calculation becomes the following:

$$\text{Slope} = (\text{Concentration}_{\text{max}} - \text{Concentration}_{\text{min}})/(\text{GC Response}_{\text{max}} - \text{GC Response}_{\text{min}})$$
$$= (3 \text{ mg/ml} - 1 \text{ mg/ml})/(3000 - 1000) = 0.001 \text{ mg/ml}$$

The concentration of the unknown can now be calculated by inserting the instrumental data of the unknown solution into the basic equation of the line, as follows:

$$\text{Concentration (y)} = \text{line slope (m)} * \text{peak area (x)} + \text{Y intercept (b)}$$
$$= 0.001 \text{ mg/ml} * 1275 + 0 = 1.275 \text{ mg/ml}$$

The percentage of the mixture that contains the compound is a ratio of the amount of substance in the sample divided by the amount of the original sample. The ratio can be of the weights, as in gravimetric quantitations, or concentrations, as in this instance. In either case, using the data from the above example, the calculation is as follows:

$$\text{Percentage} = (\text{Concentration}_{\text{substance}}/\text{Concentration}_{\text{original sample}}) * 100$$
$$= (1.275 \text{ g/ml}/3.000 \text{ g/ml}) * 100 = 42.5\%$$

Practical Application 14: Single Standard Solution

The precision and linear response of modern instrumentation can be used to further simplify the quantitation process. The quantitation process can be simplified into a single calculation using data from the unknown sample and a single reference standard. By utilizing the premise that the ratio of the sample's concentration and its instrumental response is a constant and is relative to the concentration of the substance, the quantitation calculation can be written as follows:

$$\text{Concentration}_{\text{unknown}} = (\text{Area}_{\text{unknown}} * \text{Concentration}_{\text{standard}})/\text{Area}_{\text{standard}}$$

When the calculation is performed utilizing this relationship and the data from the previous application, the same result is obtained:

$$\text{Concentration}_{\text{unknown}} = 1275 * 3 \text{ mg/ml}/3000 = 1.275 \text{ mg/ml}$$

Using an internal standard in the test solutions increases the accuracy of the results as well as introduces a quality control step into the analytical method. If the same internal standard solution is used to prepare the unknown sample and reference standard, the concentration calculation can be written as follows:

$$\text{Concentration}_{\text{unknown}} = (\text{Area}_{\text{unknown}} * \text{Area}_{\text{internal standard of standard}}$$
$$* \text{Concentration}_{\text{standard}})/(\text{Area}_{\text{standard}} * \text{Area}_{\text{internal standard of unknown}})$$

The following concentration calculation demonstrates the effect of introducing an internal standard. The only additional information required is the peak area for the internal standards for the reference standard and the unknown solutions, which were 1510 and 1525, respectively.

$$\text{Concentration}_{\text{unknown}} = (1275 * 1510 * 1 \text{ mg/ml})/(1000 * 1525)$$
$$= 1.262 \text{ mg/ml}$$

The 1% difference between the two methods is due to the use of the internal standard. The values obtained in the initial analysis were subject to variations due to sample volumes. The use of an internal standard compensates for variations in injection volumes and will provide a more accurate representation of the actual concentration of the solution.

Practical Example 25: Opinions (Knowledge and Experience)

The opinions provided by a trained clandestine lab chemist can be utilized during any portion of the investigation or prosecution of a clandestine lab operation. As previously stated, a clandestine lab chemist has knowledge beyond that of the traditional forensic chemist. Both the forensic chemist and the clandestine lab chemist must know the limitations of their training and experience and should not render opinions beyond that scope. The following are examples of the effects the opinions provided by chemists have upon various clandestine lab investigations or prosecutions.

A clandestine lab investigator in the southwestern United States came to a senior forensic drug chemist for an opinion concerning the use of lead acetate in the production of phenylacetone. The chemist responded that lead acetate could not be used to produce phenylacetone. The reaction required sodium acetate. This was a true statement in that the synthesis route of choice used sodium acetate in a reaction with phenyl acetic acid. However, he was not a clandestine lab chemist and was not aware of an alternative reaction used in the Pacific Northwest that utilized lead acetate and phenyl acetic acid to produce phenylacetone. The forensic chemist was an excellent bench chemist. However, he did not possess the knowledge and experience necessary to render the required opinion.

Practical Example 26: Opinions (Knowledge and Experience)

Two experts were providing testimony concerning a clandestine lab that was located in a remote desert location. The prosecution's witness held a B.S.

degree in chemistry and had over 10 years of experience working with clandestine drug laboratories. The defense's expert held a Ph.D. and was a respected former forensic laboratory director who published extensively. However, he had never been directly involved with a clandestine drug lab case, and his publications had done more than mention that certain types of drugs were produced in clandestine drug labs. During the verdict portion of the bench trial the judge commended the defense expert's service to forensic sciences. He then pronounced his testimony unbelievable. His opinion was valid. However, it was based on his reputation and not on his actual experience and training in the area of clandestine labs.

Practical Application 15: Opinions (Data Interpretation)

Volumes of technical information can be generated during a clandestine lab investigation. It is the job of the clandestine lab chemist to extract the relevant information and place it into some semblance of order. The following are examples of how a clandestine lab chemist brought calm to the chaos by using the physical evidence to answer some of the "who, what, when, where, why, and how" questions that required answers.

The following chemicals were found at the scene of a suspected clandestine drug lab: acetic acid, acetic anhydride, aluminum powder, bromobenzene, hydrochloric acid, mercuric chloride, methylamine, nitroethane, phenylacetic acid, and sodium acetate. The expert was asked to determine what the most likely final product of the operation was based on the chemicals found at the scene. A sequential evaluation of the potential manufacturing routes allowed the expert to establish the manufacturing route the operator most likely was implementing.

The evaluation of the chemicals begins by making a table with two columns. In one column, list the chemicals found at the scene. In the other column, list the potential products that could be manufactured and the chemical's role in the process (i.e., solvent, reagent, or precursor). The chemist should then have a table that correlates chemicals to their potential end products. Appendix C of this book was provided to be used for this purpose. The Chemistry Guide of the DEA Clandestine Laboratory Training manual is also a source of this information.

A pattern becomes apparent once all of the chemicals have been assigned a potential end product. At this point, the expert looks at individual synthesis routes to establish if all, or a significant portion, of the chemicals are present. Chemicals for multiple synthesis routes may be present. Many times, there may be chemicals present that have nothing to do with the synthesis being

Table 9.1

Chemical	Product/Route
Acetic acid	rA1 rMD1, 2, 5 pP4
Acetic anhydride	pP1
Aluminum	rA1 rM1
Bromobenzene	pPC1, 2, 3
Hydrochloric acid	rAll A, M, MD, PC
Mercuric chloride	rM1
Methylamine	pMD3, 4 pM1,4,5
Nitroethane	pA2 pMD2 pP5
Phenylacetic acid	pP1, 3, 4
Sodium acetate	rP1

Note: A = amphetamine, M = methamphetamine, MD = MDA, P = phenylacetone, PC = phencyclidine, # = synthesis route number, p = precursor, r = reagent.

used. The likely final product is the one with the most complete set of the required chemicals.

The information compiled in Table 9.1 indicates that the most probable end products from the list of chemicals are phenylacetone or methamphetamine. In one step, the expert narrowed the field of possible final products from all controlled substances that are commonly produced in clandestine labs to two.

Once the field of possible final products is narrowed to a manageable number, the expert compares the list of known chemicals to the list of precursor and reagent chemicals required for each of the various synthesis routes (Appendix C). In this example, the list of chemicals supports two different synthesis routes. One route suggests the production of phenylacetone using a phenylacetic acid, sodium acetate, and acetic anhydride. The other suggested synthesis route produces methamphetamine using phenylacetone, methylamine, aluminum, and mercuric chloride. Each method supports the existence of the other. At the time of this seizure, the manufacturing method of choice for the production of methamphetamine was a two-step process that used phenylacetone as an intermediate product.

Practical Application 16: Opinions (Data Interpretation)

The complete analysis of a reaction mixture from an operational clandestine lab produces a volume of information concerning the method the operator was utilizing to manufacture the controlled substance involved. The thorough analysis of the data from any given analytical technique may be all that is required to profile the synthesis route being used. The following is an example

Table 9.2 Reaction Mixture Components

Compound	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Mole. Wt.	Drug/Synthesis Route*
Ephedrine	58	69	79	41	59	165	M2, M3
Phenyl-2-propanone	91	134	92	43	65	134	Numerous A and M routes
1,2-Dimethyl-3-phenylaziridine	146	105	42	132	91	147	M2, M3
1-Benzyl-3-methylnaphthalene	232	217	108	215	202	232	M3, A1
1,3-Dimethyl-2-phenylnaphthalene	232	215	217	108	202	232	M3, A1

* See Appendix D.

of how the mass spectral analysis of a reaction mixture sample can be used to determine the synthesis route.

The mass spectral analysis of a clandestine lab reaction mixture produced six significant peaks in addition to the detected controlled substance, methamphetamine (Table 9.2). Each of the compounds was tentatively identified by a database search of the five most prominent ions in their mass spectrum (Appendix K). The database also indicated which manufacturing methods were associated with each compound. The pattern that emerged from the evaluation of the potential manufacturing routes indicated that the operator was converting ephedrine to methamphetamine using the hydriodic acid reduction technique.

Practical Application 17: Opinions (Production Estimates)

How much controlled substance could the operation produce is a question that will always be asked at some point during the investigation or prosecution. The production amount may or may not be an element of the crime, but it may be significant during the prosecution or the sentencing phase if a conviction is obtained.

The expert should routinely calculate the operation's estimated production as one of his opinions. The information to determine these production estimates is readily available if the lab scene was documented properly. The value that is relevant may be debatable. Is the amount of controlled substance that could be produced with the chemicals on hand the benchmark figure? Or, should the amount of finished product that could be produced if the operator had all the chemicals necessary to completely use the chemicals found at the site the appropriate value? This philosophical difference in opinion necessitates that the expert calculate a range. The following are

examples of calculations used to determine the production of various controlled substances.

The expert needs to know the amount of phenylacetone that can be produced from 1000 g of phenylacetic acid. He first calculates the reaction's conversion factor (n) by dividing the molecular weight of the phenylacetone (the product) by the molecular weight of the phenylacetic acid (the reactant). In Appendix N, the conversion factors for chemicals and drugs most commonly encountered in clandestine labs are presented.

$$\text{n} = \text{Molecular weight phenylacetone/Molecular weight phenylacetic acid} = 134/136 = 0.98$$

The weight of the phenylacetic acid (precursor chemical) is multiplied by the conversion factor to produce the weight of the phenylacetone (final product) at 100% conversion:

$$\text{Weight phenylacetone}_{\text{theoretical}} = \text{n} * \text{Weight phenylacetic acid} = 0.98 * 1000\ \text{g} = 980\ \text{g}$$

In some instances, the precursor is in a solution. The lower concentration must be accounted for in the production calculation. For example, methylamine is commonly found on a 40% (weight/volume) aqueous solution. The following example illustrates the modifications necessary to account for a diluted solution of 1000 g of a methylamine solution used as a precursor chemical.

$$\text{Weight methamphetamine HCl}_{\text{theoretical}} = \text{n} * \text{Volume}_{\text{methylamine}} * \text{Dilution factor} = 5.96 * 1000\ \text{ml} * 0.40\ \text{g/ml} = 2384\ \text{g}$$

Practical Application 18: Opinions (Production Estimates, Multistep)

Multistep reactions contain an intermediate that must be accounted for. Each step of the reaction has a conversion factor that figures into the final calculation. In these instances, the calculation boils down to a sequence of single-step calculations that use the weight of the previous calculation as the starting point for the next in the sequence. The intermediate acts as the product in one calculation, and the precursor does in the next. The calculated weight of the intermediate is used as the precursor weight for the second step of the process.

The benzyl cyanide synthesis of phenylacetone and subsequent conversion to methamphetamine HCl can be used to demonstrate the calculation sequence. The conversion factors for both steps can be calculated as in the previous examples or can be taken from a table of precalculated values:

$$n_1 = \text{Molecular weight phenylacetone/Molecular weight benzyl cyanide} = 134/117 = 1.14$$

$$n_2 = \text{Molecular weight methamphetamine HCl/Molecular weight phenylacetone} = 185/134 = 1.38$$

The weight of the phenylacetone intermediate is calculated as an independent step. Using the standard 1000 g of benzyl cyanide as a starting point, the calculation is as follows:

$$\text{Weight phenylacetone}_{\text{theoretical}} = n * \text{Weight benzyl cyanide} = 1.14 * 1000\ g = 1140\ g$$

$$\text{Weight methamphetamine HCl}_{\text{theoretical}} = n_2 * \text{Weight phenylacetone}_{\text{theoretical}} = 1.38 * 1140\ g = 1573\ g$$

Practical Application 19: Opinions (Per Batch Production Estimates)

The per batch estimate can be a significant point of debate. The potential of an operation to produce 10 kg of controlled substance loses its significance if it can only be produced in 10 g batches due to limitations placed upon it by the size of the available equipment. The following is an example of estimating the per batch production of an operation, using the equipment as a limiting factor.

The expert is asked to calculate the amount of methamphetamine that could be produced using a 1000 ml reaction flask. Without additional information, the expert makes the following assumptions. First, he uses a common chemical ratio for the methamphetamine reaction, which is 4 l of acid, 1 kg precursor, and 500 g of an additional reagent. He also assumes a reaction mixture volume of 2/3 the total volume of the reaction flask. His calculations are as follows:

$$\text{Volume}_{\text{reaction mixture}} = \text{Volume}_{\text{flask}} * 66\% = 1000\ ml * 0.66 = 660\ ml$$

$$\text{Reaction ratio} = \text{Precursor amount/Acid amount}$$
$$= 1000 \text{ g}/4000 \text{ ml} = 0.25 \text{ g/ml}$$

$$\text{Weight precursor}_{\text{reaction mixture}} = \text{Reaction ratio} * \text{Volume}_{\text{reaction mixture}}$$
$$= 0.25 \text{ g/ml} * 660 \text{ ml} = 165 \text{ g}$$

$$\text{Weight product}_{\text{theoretical}} = n * \text{Weight precursor}_{\text{reaction mixture}}$$
$$= 0.92 * 165 \text{ g} = 152 \text{ g}$$

Practical Example 27: Testimony

The following is an example of the need for understanding the definitions of the terms that are used during the testimony. The original exchange was between a defense attorney and the State's expert during a controlled substance trial. This exchange could easily have happened between an attorney and his own expert without pretrial preparation.

Attorney: Mr. Chemist, did you perform a qualitative analysis on Exhibit A?
Chemist: Yes sir.
Attorney: What percentage of Exhibit A contained a controlled substance?
Chemist: I did not perform that examination.
Attorney: Did you perform a qualitative analysis on Exhibit A?
Chemist: Yes sir.
Attorney: And what percentage of Exhibit A contained a controlled substance?
Chemist: I did not perform that examination.
Attorney: Mr. Chemist, you said you performed a qualitative analysis on Exhibit A?
Chemist: Yes sir.
Attorney: Then what percentage of Exhibit A contained a controlled substance?
Chemist: I did not perform that examination.
Attorney: Mr. Chemist, why won't you answer my question?
Chemist: Sir, I am trying to use proper terminology, as you requested.

This exchange demonstrates that the attorney clearly did not understand the difference between qualitative (what is it?) analysis and quantitative (how much is there?) analysis. The chemist's responses clearly created an adversarial atmosphere by specifically answering the question posed. He could have

provided the information the attorney desired in a less combative style, placing him in a better light with the jury.

Practical Example 28: Testimony

The chemist analyzed a 10 bail representative sample of a 200 bail seizure of marijuana. The pretrial preparation consisted of brief introductions. Below is an excerpt of the resulting testimony.

Attorney: Mr. Chemist, I show you Exhibit A. Do you recognize it?
Chemist: No sir, I do not. (The chemist was presented a 50-lb, plastic-wrapped bail of plant material. He looked and did not find the identifying marks he placed on it at the time of analysis. He expected to be presented one of the 10 bails he actually analyzed.)
Attorney: **Silence.** (The attorney expected a Yes, which would lead into his next question.)
Attorney: Do you recognize anything on Exhibit A?
Chemist: Yes sir, I recognize the case number.
Attorney: Where do you recognize that case number?
Chemist: From a submission I analyzed in July of this year.
Attorney: What did that submission consist of?
Chemist: Ten plastic wrapped bails of plant material.
Attorney: Did those exhibits resemble Exhibit A?
Chemist: Yes sir.
Attorney: Did you analyze the exhibits submitted to you in July of this year?

The testimony continued. The results of the analyses of the representative samples were eventually allowed into the record.

This whole exchange could have been avoided and boiled into a few flowing questions. Do you recognize the exhibit and how? Did you analyze the exhibit? What are the results of your analysis? However, the lack of proper preparation resulted in both parties being surprised, resulting in additional questions and answers to establish the same facts.