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# Edward G. Brown, Ph.D.

Dr. Brown obtained his Bachelor of Science degree in Chemistry from U.C. Berkeley in 1980 and his Doctoral degree in Organic Chemistry from U.C. Davis in 1988. Two post-doctoral chemistry research fellowships from University of Auckland, New Zealand, and Yale University furthered his academic studies in medicinal chemistry and analytical chemistry techniques through 1990.

Dr. Brown's productivity during his career as a medicinal and organic chemistry researcher in academics and while working at several pharmaceutical companies and other laboratories has led to his co-inventorship on ten US patents and his co-authorship on over thirty research articles and conference presentations.

Dr. Brown is also a patent agent who specializes in chemistry patent drafting and assists in patent litigation and prosecution issues for patent law firm clients in North Carolina and around the US.

In 1991, Dr. Brown began his consulting work as an expert witness in an LSD case. He has since continued to develop his expert witness practice throughout the intervening years, and to date, has assisted nearly 100 attorneys in law firms from North Carolina, Virginia, Maryland, Pennsylvania, and many other States, as well as in Canada and Great Britain on a large variety of drug cases and DWI/DUI cases, both at the State and Federal levels. He has testified in criminal drug cases and DWI/DUI cases as a Chemistry Expert in both Federal and State courts around the United States and has been deposed as a Chemistry Expert in a patent litigation case.

# Confirmatory Drug Analysis: Methodology Used and Common Errors

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# Today's Agenda

## 1. Brief Summary of Presumptive Tests and Analysis Methods.

Techniques for Initial Detection of Illegal Drugs and Related Materials:

- A. Color Tests, Microcrystalline Tests, Elisa Tests, UV and IR spectroscopy.
- B. How and why they are used.

## 2. Confirmatory Test Methods.

Instrumentation Used for Drug Sample Analysis and Chemical Identification:

- A. FTIR, GCMS and LCMS Analysis Methods.
- B. How and why these are used.
- C. False-positives and false-negatives from these methods.
- D. Short Questions & Answers break.

## 3. Specific Issues Related to Drug Testing and DUI/ DWI.

Misassumptions or Mistakes by Chemists, Attorneys and Police Officers:

- A. Assuming that Scientists Never Make Incorrect Assumptions or Mistakes.
- B. Assuming that Presence of a Drug in the Bloodstream Means Impairment.
- C. Allowing the Expertise of a Witness to Expand into Areas of Non-Expertise.
- D. Questions & Answers.

# Presumptive Tests vs Confirmatory Tests in Drug Evidence Testing & Analytical Chemistry:

What Are Presumptive Tests? What Are Confirmatory Tests? How and Why Are Each Type Used? How Do These Analysis Methods/Classifications Differ?

## Presumptive Test Methods:

In analytical and forensic sciences, a presumptive test is an analysis method which is used initially to quickly and easily establish:

If a chemical sample of interest contains no substances which give positive test results. Negative test results quickly rule out the need for further testing of the sample using more expensive or more time-consuming methods.

Or, if a chemical sample gives a positive test result, this indicates that the sample may contain one or more substances that require further analytical work to be performed.

## Confirmatory Test Methods:

Tests that are required to confirm what chemicals are present in a sample are referred to as confirmatory tests. These tend to cost more than simple presumptive tests, take more time to perform & review, are far more accurate and have fewer false positives.

# Presumptive Tests: Methods, Benefits & Limitations:

## Examples of Presumptive Test Methods:

Examples of presumptive tests include color tests/spot tests, microcrystalline tests, ultraviolet spectroscopy (UV), infrared spectroscopy (IR), microscopic examinations, enzyme-linked immunosorbent assays (ELISAs) and thin layer chromatography (TLC).

## Presumptive Test Benefits:

- Presumptive tests are relatively inexpensive & they are rapidly and easily performed.
- They can help to narrow or eliminate the need for further evidence sample testing.
- Many of these can help an analyst decide which analytical tests to perform next.
- Many are fairly sensitive & can be employed to detect various chemical & drug types.

## Presumptive Test Limitations:

- Tests have higher risk of false positives or interpretations that lead to false positives.
- Cannot conclusively identify a substance; can only hint at identity possibilities.
- Most methods require the destruction of a small amount of an evidence sample.
- Require the use of chemical reagents which are hazardous to skin and eyes.
- Some of the test reagents require hazardous waste disposal methods to be employed.

# Presumptive Tests: False Positives & Negatives:

## Sources of False Positive & False Negative Results with Presumptive Tests:

- IR Spectroscopic Analyses:
- In the case of some seized drugs, the complexity of the matrices (# of cutting agents) and the low percentage of drug in the evidence sample compared to the amount of matrix present makes qualitative tests especially prone to produce false negatives.
- The enantiomeric D- and L-isomeric forms of drugs will not be differentiated when using IR spectroscopy. In the case of methamphetamine, the L-isomer is legal to buy, sell and possess and one can purchase products made with this isomer at drug stores over-the-counter. An analysis by IR or FTIR will not prove that the illegal isomer is present, only that some type of methamphetamine is present.
- IR spectroscopic analysis of samples can give different results for the IR spectra from the same chemical when in different salt forms, in other words, if the pH of the chemical matrix is either acidic or basic. These IR spectral changes must be kept in mind when analyzing the spectra from evidence samples.
- If an evidence sample contains many different chemicals, such that many of the absorbances are broadened by the overlapping of peaks and troughs from the impurities, it may be possible to mistake the peak pattern for that of a drug.

# Challenging admissibility of field drug test results: Examples of Case Law:

- *State v. Ward*, 364 N.C. 133 (2010) – visual inspection not enough, chemical test needed for an expert to testify to the identify of a substance
- *State v. Carter*, 237 N.C. App. 274 (2014)
- *State v. Pinnix*, 246 N.C. App. 190 (2016)
- *State v. Cobb*, 845 S.E.2d 870 (2020)
- *State v. Osborne*, 372 N.C. 619 (2020) “To be sure, much of the State’s evidence identifying that rock-like substance as heroin, such as the field test results, might have been excluded had Osborne objected.”

# Weight

- Net weight = weight of item without packaging
- Gross weight = weight of item with packaging
- Will report net weight unless it is not possible to separate item from packaging. See [Technical Procedure for Balances-Drug Chemistry](#).
- Will give uncertainty budget and level of confidence. These refer only to confidence in weight, not in identification of the drug.
- Lab report will specify weight received and weight returned in the “case notes.”

One plastic bag was analyzed and found to contain:

Methamphetamine - Schedule II.

Net weight of material - 0.53 (+/- 0.06) gram.

Analysis of the above item was conducted using the following methods: color test, IR.

Measurement uncertainty of reported net weights is at a 99.7% level of confidence.

# Sampling

- If the entire item of evidence was not tested and a sampling plan was used to determine what portion to test, it will be described in the “case notes.”
- There are 3 types of sampling plans:
  - Administrative – test one item (for pills)
  - Threshold – test up to a threshold weight
  - Hypergeometric – test a statistically-determined number of items and make an inference about the rest

See [Administrative Procedure for Sampling](#)

*Consider – is the sampling plan a reliable method for testing a representative portion of the evidence? If not, make a 702 challenge.*

# Confirmatory Tests: Methods, Benefits & Limits:

## Examples of Confirmatory Test Methods:

Examples of confirmatory tests include infrared spectroscopy (IR & FTIR), gas chromatography-infrared spectroscopy (GCIR), gas chromatography-mass spectrometry (GCMS), liquid chromatography-mass spectrometry (LCMS) and Nuclear Magnetic Resonance (NMR).

## Confirmatory Test Benefits:

- Confirmatory tests are relatively accurate and can conclusively identify a substance;
- The tests have a much smaller risk of creating false positives than presumptive tests;
- When chromatographic methods such as GCMS or LCMS are used in a method, the chemical components in an evidence sample can be separated from one another prior to being analyzed; the purified components are analyzed by the second analysis method and the tandem-method provides a greater level of surety in the test results.

## Confirmatory Test Limitations:

- Methods require the purchase and upkeep of expensive equipment.
- Confirmatory methods require more time and training to use the instrumentation.

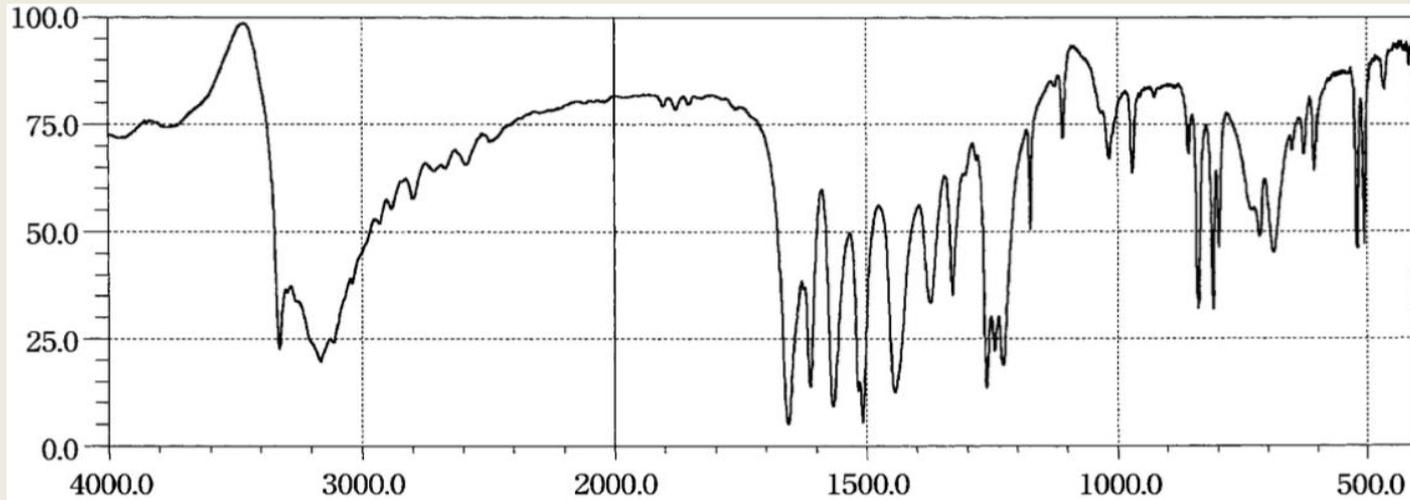
# Confirmatory Tests: Infrared Spectroscopy (IR)<sup>1</sup>

## Infrared Spectroscopy (IR) and Fourier-Transformed IR (FTIR): How It Works:

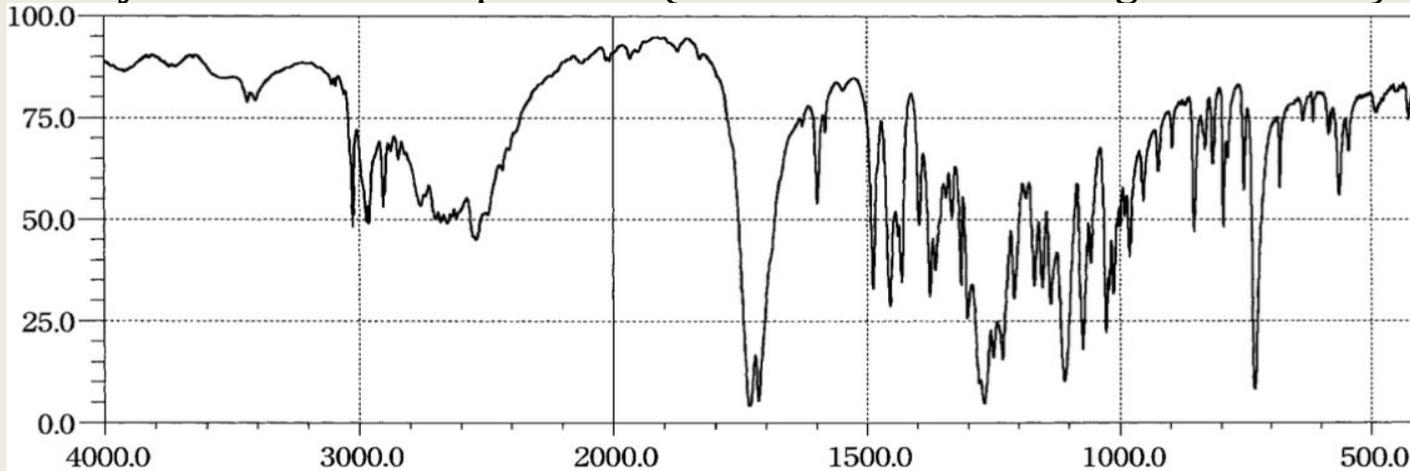
- Infrared (IR) spectroscopy is another highly discriminatory method and is based on the measurement of the amount of IR radiation which is absorbed or emitted by a sample as a function of the IR light wavelength passing through the sample.
- We sense light in this IR range as heat; the frequencies of IR radiation correspond with the vibrational frequencies between the atoms and groups in molecules.
- A spectrum is obtained by passing infrared radiation through a sample and determining the amount of the incident radiation (radiation that actually hits the molecule rather than passing through) that is absorbed at each IR frequency.
- The vibrational structure of a whole molecule determines what IR wavelength(s) are absorbed versus which pass through a sample.
- Interpretation of the spectra allows for determination of the types of molecular functional groups present in a sample.
- The IR spectrum of a pure compound provides a distinctive fingerprint which can be differentiated from the IR absorption pattern of other compounds.
- Drugs can be identified through databases (such as at <http://webbook.nist.gov/> )
- NC State Crime Lab Procedure now requires the identification of at least 6 IR peaks (wavelengths) in an IR spectrum & comparison of those peaks with a standard sample.
- Examples of IR spectrophotometric analysis spectra are given on the next slide.

# Confirmatory Tests: IR Spectroscopy Examples:<sup>24</sup>

Acetaminophen IR Spectrum (But Note No Wavelengths Marked):



Cocaine Hydrochloride IR Spectrum (But Note No Wavelengths Marked):



- 5.4.3.2** An IR spectrum of a controlled substance shall compare favorably to the IR spectrum of a known reference standard before an identification is confirmed.
- 5.4.3.3** When using FTIR as the primary structural elucidation technique, the sample spectrum shall compare favorably with a spectrum of a known standard in both its overall appearance and in the presence and location of major peaks.

**5.4.2.2** Compare the unknown spectrum to a known reference material.

**5.4.2.2.1** Six prominent and well-defined peaks in the sample spectrum between  $2000\text{ cm}^{-1}$  to  $650\text{ cm}^{-1}$  shall be labeled. The same six peaks shall be present within  $\pm 1\text{ cm}^{-1}$  of those in the reference spectrum during comparison for identification.

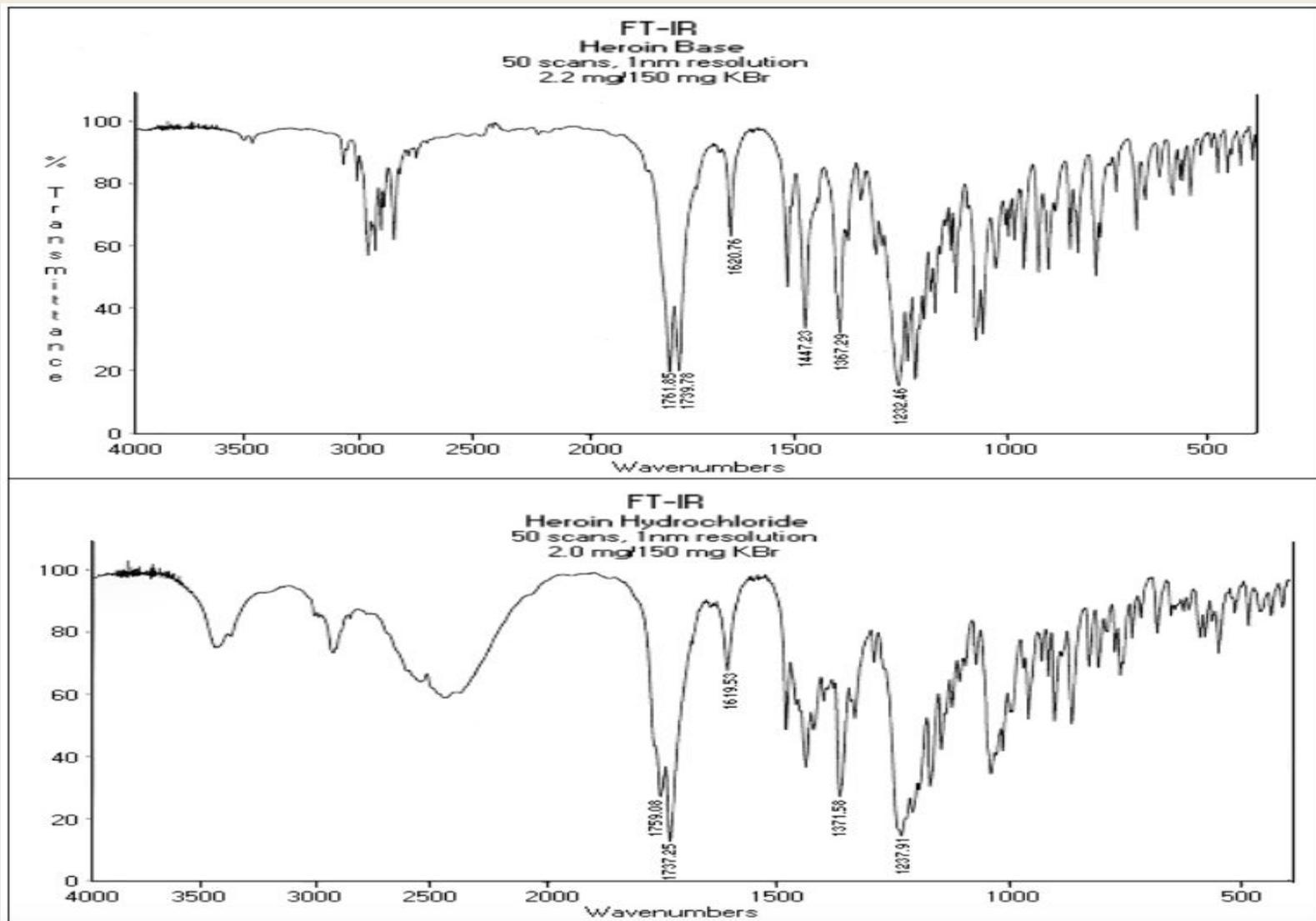
- If there are less than six prominent and well-defined peaks in the sample spectrum between  $2000\text{ cm}^{-1}$  to  $650\text{ cm}^{-1}$  then all peaks shall be present within  $\pm 1\text{ cm}^{-1}$  of those in the reference spectrum during comparison.

**5.4.2.2.2** The overall spectral pattern shall correspond to that of the reference material in regards to the absence or presence of major peaks and relative peak intensities.

**5.4.2.2.3** No prominent unexplainable extraneous peaks shall be observed in the sample spectrum.

# Confirmatory Tests: IR Spectroscopy Examples:<sup>25</sup>

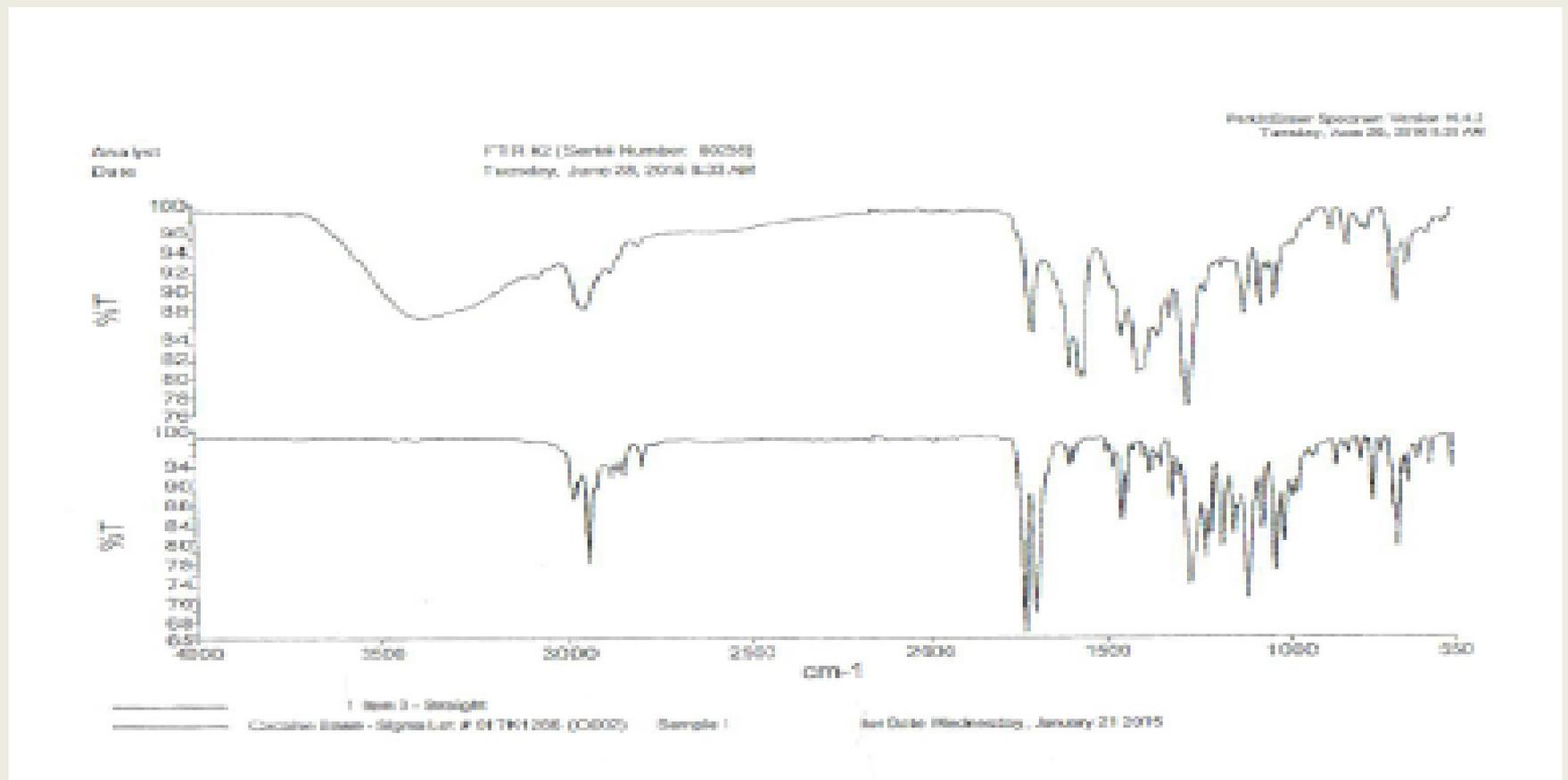
Heroin Free Base & Hydrochloride Salt (Note Wavelengths Marked In These):



# IR Presumptive Tests vs Confirmatory Tests: IR Spectroscopy Example from Cocaine Case:

Top IR Spectrum: Evidence Sample (Note No Wavelengths Marked Here)

Bottom IR Spectrum: Cocaine Base Standard (Note No Wavelengths Marked Here)



# Confirmatory Tests: IR and FTIR:<sup>30</sup>

## Infrared (IR) Spectroscopy: How It Works:

- As stated in the previous section, IR spectroscopy measures the amount of infrared light absorbed by a sample at many different wavelengths of IR light; the spectrometric technique measures the absorbance of light at wavelengths between  $\sim 2.5\text{-}25\ \mu\text{m}$ ; in essence, the technique measures the types of vibrational energies (a type of heat energy) that can be absorbed by a chemical sample;
- When IR spectra show many very sharp absorption peaks (or troughs) in the spectra which can be compared easily to the IR spectra from other known standard samples of a drug or chemical, the method can be useful for confirming the identity of a drug.
- The difference between the use of IR spectroscopy as a presumptive technique and its use as a confirmatory technique is subjective; it relies upon the purity of the sample analyzed, the wavelength resolution of the instrument, the reliability of the chemical standard used for a comparison with an evidence sample, and the training of the operator. Pure samples, more reliable comparison samples and greater operator training point to its use as a confirmatory method whereas very impure samples coupled to a lack of reliable standard samples for comparisons, less wavelength resolution and lower operator know-how make its use more presumptive.
- In addition, NC State Crime Lab procedure now requires the use of wavelength annotation in confirmatory tests employing IR or FTIR (6 peaks minimum).

# IR Spectroscopy: False Positives & Negatives:

## Sources of False Positive & False Negative Results with IR Analyses:

### IR Spectroscopic Analyses:

- In the case of some seized drugs, the complexity of the matrices (# of cutting agents) and the low percentage of drug in the evidence sample compared to the amount of matrix present makes these tests especially prone to produce false negatives.
- The enantiomeric D- and L-isomeric forms of drugs will not be differentiated when using IR spectroscopy. In the case of methamphetamine, the L-isomer is legal to buy, sell and possess and one can purchase products made with this isomer at drug stores over-the-counter. An analysis by IR or FTIR will not prove that the illegal isomer is present, only that some type of methamphetamine is present.
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- If an evidence sample contains many different chemicals, such that many of the absorbances are broadened by the overlapping of peaks and troughs from the impurities, it may be possible to mistake the peak pattern for that of a drug.

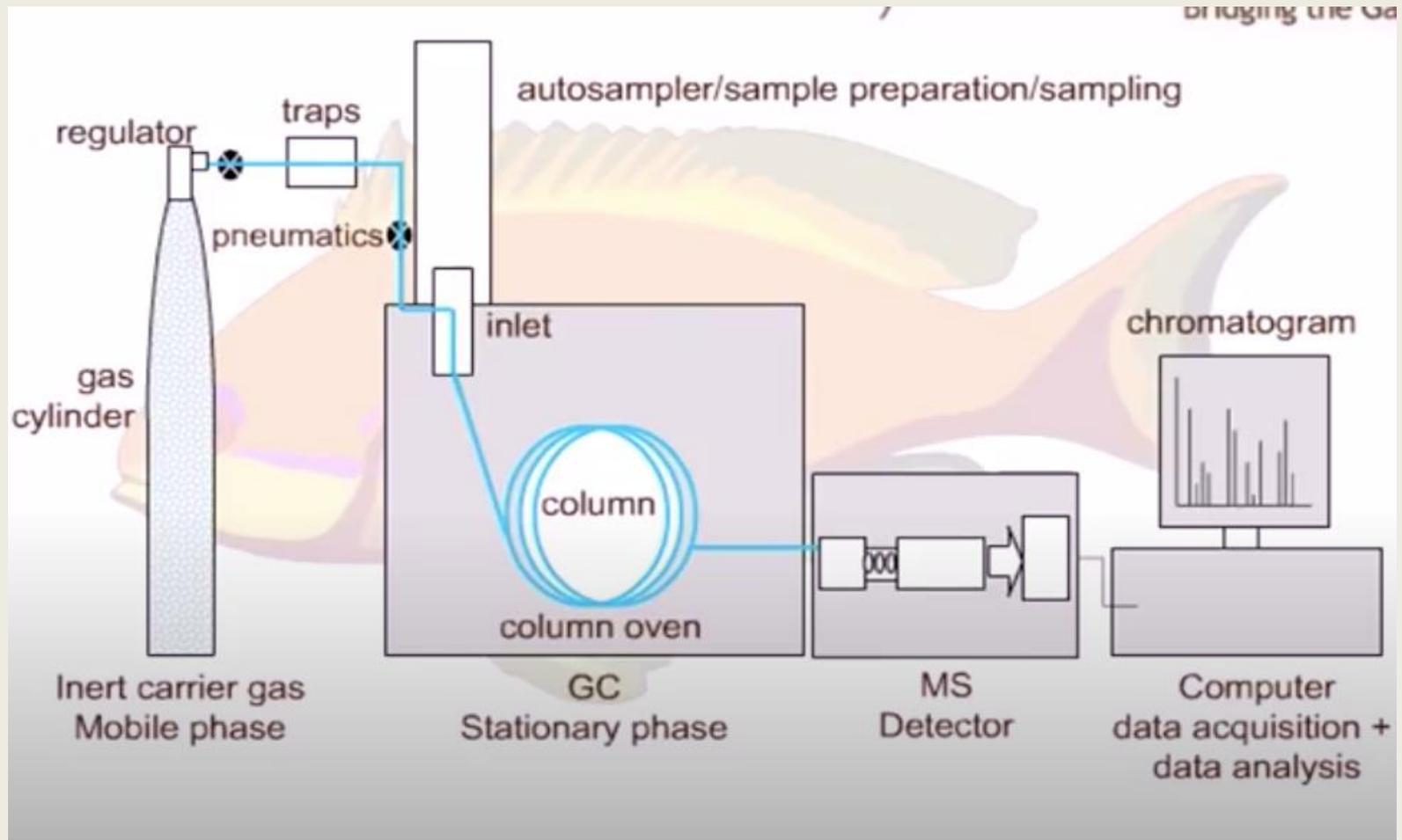
# Confirmatory Tests: GCMS:<sup>1,31</sup>

## GCMS: How It Works:

- Gas chromatography (GC) is a technique used to separate chemicals in a mixture using each chemical's vaporization tendency versus its attraction to a porous solid support coating the inside a long capillary tube; a gas such as helium, while under high pressure, is passed through a capillary column in the instrument. The column is located in an oven in which the temperature can be heated gradually. A sample is injected into a very hot injection port and is swept into the flow of gas into the front end of the capillary column. The different components in a mixture are moved forward through the column to some degree by the gas flow but are also retained by a degree of attraction to the solid support in some way; the differences in the retentive forces vs the vaporization rates and flow of each component as a vapor through the capillary lead to the separation of the components. Each component leaves the other end of the capillary at some point and is detected by a detector such as a "Mass Spec".
- Mass Spectrometry (MS) is a method that can be used to detect chemicals as they come off of a GC capillary column. Each analyte enters the MS and is subjected to an ionizing form of particles which causes the molecules of the analyte to fragment and ionize. Each of the fragment ions has a mass that can be separated from the other masses and can be counted. The MS instrument tallies each mass fragment and creates a pattern on a graph of the masses. This acts like a fingerprint for the drug.

# Confirmatory Tests: GCMS:<sup>31</sup>

## GCMS: How It Works:

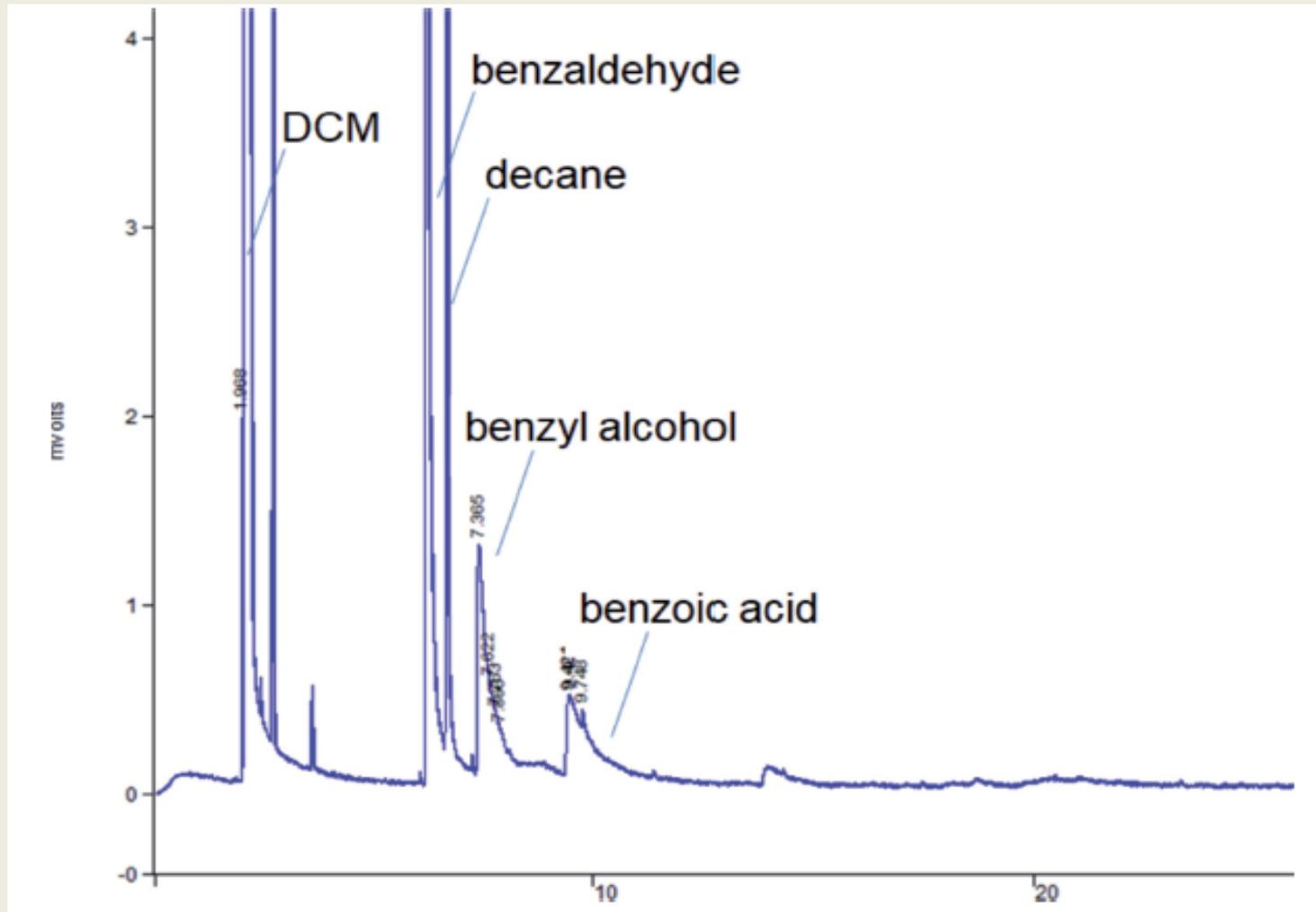


# Chromatography: The Shopping Mall Analogy:



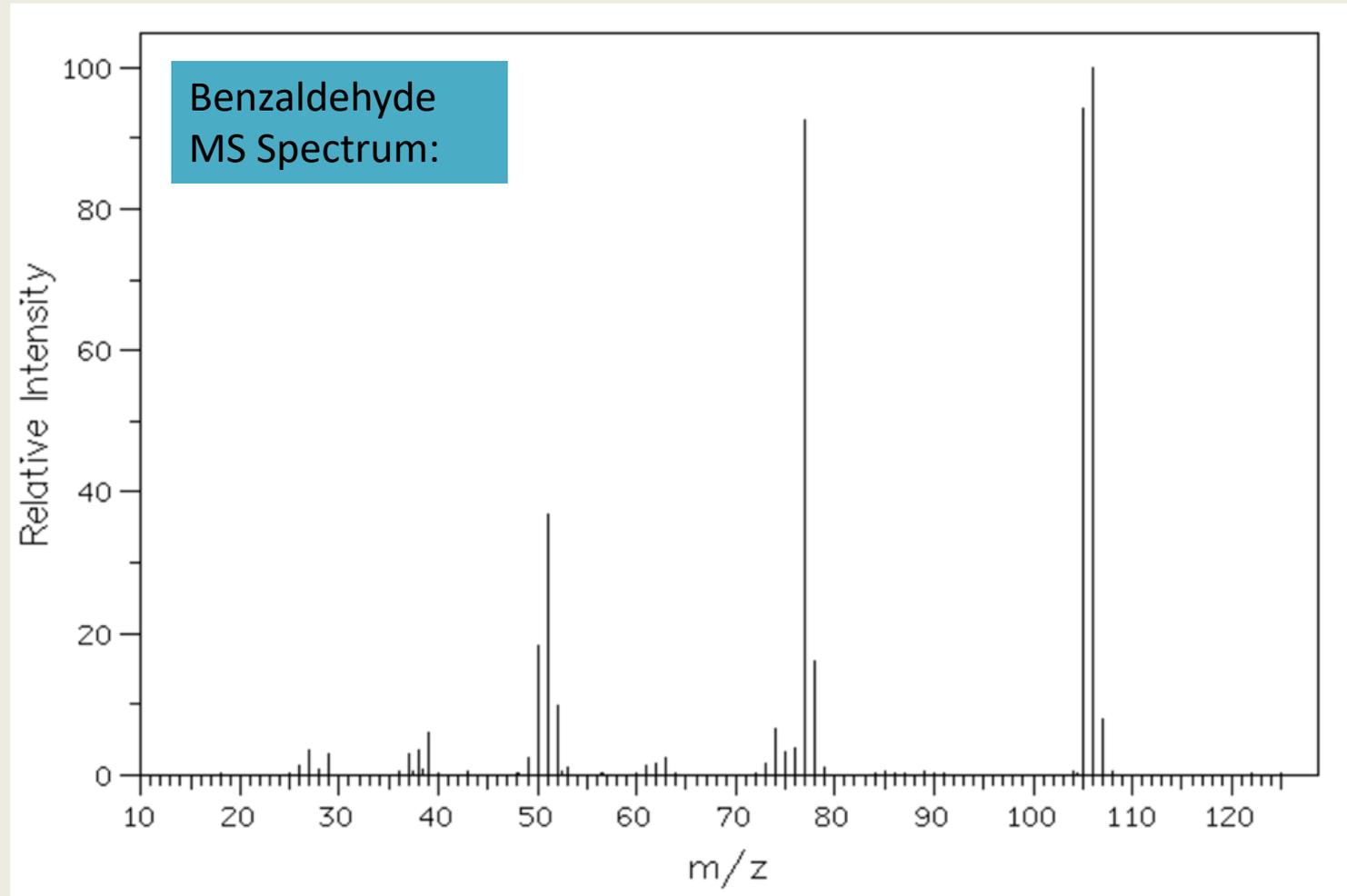
# Confirmatory Tests: The GC Printout from GCMS:<sup>32</sup>

GCMS: Example of Chromatogram from a Mixture of Chemicals:



# Confirmatory Tests: The MS Printout from GCMS:<sup>35</sup>

- GCMS: Example of Mass Spectrum from One Chemical in a Mixture:



# Confirmatory Tests: LCMS:<sup>33</sup>

## LCMS: How It Works:

- Liquid chromatography (LC) is very similar to GC, except that a flowing liquid is used to separate chemicals in a mixture instead of a flowing gas. Each chemical's solubility properties are pitted against its attractiveness to a porous solid support packed into a steel column. Separation of a mixture occurs along the length of the column.

While a liquid solvent mixture under high pressure is passed through the column of the instrument, a sample is injected into the flow of solvent; this material is swept into the front end of the column and the mixture moves forward over time.

The different components in a mixture are moved forward through the column to different degrees by their ability to dissolve in the flow of solvent but are also retained by their degree of attraction to the solid support.

The differences in the retentive forces vs the solubility and flow of each component through the column lead to the separation of the components. Each component leaves the end of the column during the sample run and is detected by a detector such as a "Mass Spec" (MS).

- Mass Spectrometry (MS) is used as the detection method in LCMS in a similar way as in GCMS.

# Confirmatory Tests: False Positives & Negatives:

## Sources of False Positives & False Negatives with Confirmatory Tests:

Confirmatory tests measure or provide evidence of the chemical structures of the chemicals within evidence samples. Since these methods provide direct measurements about the actual molecules present in a sample, other chemicals, impurities or additives do not interfere with the testing to the same degree that can occur in presumptive tests. Fewer false-positives and false-negatives can occur using confirmatory tests than when using presumptive testing methods.

### - GCMS:

- GCMS is one of the most common methods used for testing of evidence samples. It is extremely sensitive and can detect traces of contaminants in a sample at levels of a billionth of a gram (nanograms) or less; false positives can occur, therefore, if care is not used when preparing multiple samples while using the same gloves.
- GCMS requires the use of a very hot injection port to vaporize the contents of each sample as it is injected into the column. Sometimes this elevated temperature can lead to sample decomposition and can create an illegal drug from a legal substance through the decomposition process. Sudafed is such a drug– it decomposes to form methamphetamine under certain conditions and it can give a false-positive.

# Confirmatory Tests: False Positives & Negatives:

## False Positives & False Negatives with Confirmatory Tests, Continued:

- GCMS, cont'd:
- Unless a special type of chiral-phase capillary column is used to separate the components in a mixture, GCMS will not separate D- and L- isomers of drugs or other D-/L-chemicals. This means that under normal circumstances, the legal L-isomer of methamphetamine (called Levmetamfetamine in over-the-counter products at the drug store and in FDA literature) will exit the GC column and be detected by the MS instrument at the same time as the illegal D-isomer of methamphetamine. This can lead to false positives from blood or urine samples taken from drivers who have been using these decongestant cold medications while driving.
- Because the GCMS technique is so sensitive, it is important to show that the GCMS system is clean and free of residual contaminants; these may have been left on a dirty injector needle or may still be present from a previous analysis sample run on the capillary column. GCMS instrument programs need to run a solvent blank between evidence samples and/or control samples in order to rule out the possibility that a peak in a GCMS trace is a false-positive from a dirty instrument.

# Confirmatory Tests: False Positives & Negatives:

## False Positives & False Negatives with Confirmatory Tests, Continued:

- GCMS, cont'd:
- It is important that the blanks run between samples contain solvent, rather than just air. An air blank will give the perception that a blank has been run between samples, but air will not remove any residue still present in a contaminated injector needle, so it could lead to a false-negative for the blank and a false-positive for the next sample run. Running a blank using an injection of air is like trying to shower using a stream of air– it doesn't serve any purpose.
- The GCMS technique usually involves the analysis of many unrelated samples on the same day using an automated device on a GCMS instrument; the device that holds all the samples is usually referred to as an autosampler. It is important that an analyst or group of analysts follow proper lab protocols to decrease the possibility of human error. This can occur by mislabeling of evidence sample vials or through incorrect placement of each GCMS sample vial in the proper numbered slot on the autosampler. The sample ID that is entered into the GCMS computer should be double-checked to make sure that the vial selected for GCMS analysis by the computer program is the same vial that is present in the numbered position.

# Confirmatory Tests: False Positives & Negatives:

## False Positives & False Negatives with Confirmatory Tests, Continued:

- LCMS:
- The issues that can produce false-positives and false-negatives with the LCMS technique are very similar to those listed for the GCMS technique, with the exception that thermal decomposition by the injector port of an LC instrument does not occur. Although the injector port of a gas chromatography instrument is heated to promote vaporization of the samples, the injector ports of liquid chromatography instruments are not heated to high temperatures, since vaporization of the samples does not play a role in LCMS instrumentation techniques.
- LCMS techniques involving reverse-phase liquid chromatography usually employ solvent mixtures that contain small amounts of acid. In rare circumstances, the acid mixtures in these solvents employed for the chromatographic purification can lead to small amounts of sample decomposition during the chromatography, possibly with the creation of new products that have similar or identical masses as illegal drugs. These can leads to false positives in the detector.

# Should I make a 702 challenge?

## Considerations -

- Analyst qualifications:
  - Is the analyst certified in Drug Analysis? Does the analyst have sufficient training and experience?
- Reliability of the method as a whole:
  - Is the sample a mixture? Were the peaks of the FTIR labeled? (At least 6 peaks labeled and compared with a standard sample's same 6 peaks within +/- 1  $\text{cm}^{-1}$ ?)
- Reliability of the method as applied in this case:
  - Was appropriate sampling used? Is the sample a mixture? Is the substance an optical isomer of a legal substance (D and L meth) and was that proven by a chemical analysis rather than by assumption? How much time was spent on the entire analysis? Were the peaks of the FTIR labeled?

# Confirmatory Tests: False Positives & Negatives:

Questions and Answers:

???

# Specific Issues Related to Drug Testing & DUI/DWI:

## Misassumptions or Mistakes by Chemists, Attorneys and Police Officers:

### A. Assuming that Scientists Never Make Incorrect Assumptions or Mistakes:

- Do not do this! Scientists are human and make occasional mistakes. Some scientists think that they are superhuman and do not admit to making mistakes, but they must be able to prove that the equipment they used were working accurately on the day they used them. Require that all proof of accurate weights and standards used are documented & present in the courtroom as written documents. Don't allow verbal testimony based upon memory to suffice for this.

### B. Assuming that Presence of a Drug in the Bloodstream Means Impairment:

- Do not do this! Usually, the amount of a drug detected in a blood sample is not quantified at all by many State Labs. This makes it impossible to tell if the drug was present at a level that would be impairing, without other expert testimony from a person trained to determine impairment and who knows what blood concentrations of a drug are considered impairing. Do not allow a witness to testify on drug levels and impairment if they are not experts in those areas.

### C. Allowing the Expertise of a Witness to Expand into Areas of Non-Expertise:

- Do not do this! Human beings have opinions on all matters in life; Experts are no different. Many enjoy opining on other related subjects where the Voir Dire process did not cover or show their expertise in those subjects.

# Summary:

My intention has been to describe the basics about various analytical tools and techniques used by forensic analysts when determining if drugs or alcohol are present.

I hope you have a better understanding of these issues now.

Please remember that there are many types of data that need to be presented in order to prove to a level of scientific certainty that drugs or alcohol are present in a person's body. It takes much less evidence, however, to make a presumptive guess about the presence or absence of drugs or alcohol.

In many cases, questions need to be asked about subtle assumptions and data differences in the evidence; without a detailed knowledge an Expert has about chemistry and toxicology, these questions might not be asked at the appropriate times; this matters.

Drug and alcohol chemistry topics can be complicated, but an understanding of these issues doesn't have to be impossible. I would recommend finding an Expert who can help you to understand the details of the science in each of your cases.

We can provide expertise in chemistry and toxicology issues.

Our First Hour of Work is Free!

Thank You for Your Time and Attention!

Thank you!!!

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<https://www.ncbi.nlm.nih.gov/pmc/articles/PMC5537996/> : Accessed 6/14/2020.
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[http://swgdrug.org/Documents/SWGDRUG%20Recommendations%20Version%208\\_FINAL\\_ForPosting\\_092919.pdf](http://swgdrug.org/Documents/SWGDRUG%20Recommendations%20Version%208_FINAL_ForPosting_092919.pdf) : Accessed 6/14/2020.
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<https://www.ncjrs.gov/pdffiles1/nij/183258.pdf> : Accessed 6/14/2020.
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6. For Duquenois–Levine reagent, see: [https://en.wikipedia.org/wiki/Duquenois–Levine\\_reagent#/media/File:Duquenois\\_levine\\_step2.jpg](https://en.wikipedia.org/wiki/Duquenois–Levine_reagent#/media/File:Duquenois_levine_step2.jpg) : Accessed 6/14/2020.
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