

Analysis

R201119281

Item #1 submitted on November 7, 2011 by Sheleathea Wright-Quick of the Chapel Hill Police Department in lab number R201119281 described as a "Plastic bag containing pieces of clear capsule and off-white powder material". The results of the examination are reported in this report as:

RESULTS OF EXAMINATION:

Item 1

Off-white powder material:

2,5-dimethoxy-4-ethylphenethylamine (2C-E).

Weight of material - less than 0.1 gram.

Capsule pieces - No analysis.

On page 12 of 20 of this report, Attachment A, the analyst advises that a Marquis test was utilized and the resultant color found was "yellow". The passage below is from the protocol published on-line by the NC State Crime Lab. Note that the colors found for this reagent with particular types of materials do not include the color yellow. The closest material in chemical structure to the material reported as found, 2,5-dimethoxy-4-ethylphenethylamine (2C-E) is amphetamine and the color from a Marquis test with that material is "orange/brown." This is then a negative test, not consistent with the presence of those materials the NC Crime Lab can and should be tested with this test. The fact that the color seen was yellow means that the analyst has no idea what kind of material she has based upon this test.

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Technical Procedure for Preliminary Color Tests

1.0 Purpose - This procedure specifies the required elements for the preparation and use of preliminary color test reagents.

2.0 Scope - This procedure applies to all preliminary color tests used in the Drug Chemistry Sections of the State Crime Laboratory.

3.0 Definitions

- **Prepared reagent** – Mixture of two or more reagents or a dilution.
- **Commercial reagent** – Solvent or chemical purchased from an outside vendor.
- **Performance verification** – The initial confirmation of the reliability of a previously or externally validated method or instrument.
- **Quality control (QC) checks** – Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.
- **Reference material** – Material sufficiently homogenous and stable, with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties.

4.0 Equipment, Materials and Reagents

4.1 Equipment

- Balance

4.2 Materials and Reagents

- Fume hood
- Eye protection
- Laboratory coat
- Gloves
- Beakers or other glass vessels (Optional)
- Culture tubes (6 X 50 mm suggested size)
- Funnel
- Glass stirring rod
- Graduated cylinder
- Pipettes with bulb
- Porcelain spot plates (Black suggested for Barium Chloride Reagent)
- Reagent bottles and stock bottles (amber-colored preferred for Duquenois reagent)
- Spatula
- Weigh boats or other weigh vessels
- Filter paper (PDMAB and Koppanyi only)
- Scissors (PDMAB and Koppanyi paper only)
- Wide mouth bottles with tops (suggested for storage of PDMAB and Koppanyi Paper)
- Commercial Reagents
- Reference materials

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5.0 Procedure

5.1 Standards and Controls – Quality control checks of all reagents shall consist of a negative check and a positive check. Both checks shall be acceptable according to the procedure listed for each reagent, and shall be recorded together as a quality control check in the Resource Manager section of FA.

5.1.1 Negative quality control checks shall be performed according to the procedure listed with no sample present.

5.1.1.1 Acceptable result is no significant color formation.

5.1.1.2 If a significant color develops, steps shall be taken to ensure the spot plate is clean. Making new reagent and retesting with no sample present are further steps that can be taken to ensure no significant color develops prior to introduction of the sample.

5.1.2 Positive quality control checks shall be performed according to the procedure listed for each reagent using the specified reference material. See each procedure for acceptable results.

5.1.2.1 The result of the quality control check shall be recorded in FA with the identification of the standard used and the results of the QC check.

5.1.3 Reagents may be prepared in any amount provided that the component ratios are kept constant.

5.1.4 Labeling - All stock and use bottles shall be labeled according to the Administrative Policy for Drug Chemistry Quality Assurance.

5.1.5 Storage - Stock and use solutions shall be stored in closed containers. All stock bottles shall be stored in the refrigerator, and all use bottles kept on the countertop or under the hood, unless otherwise noted in the procedure. All bottles shall be labeled as described in the Administrative Policy for Drug Chemistry Quality Assurance.

5.1.5.1 Expiration Dates - Stock bottles stored in the refrigerator have a three year expiration date. They shall be labeled as such.

5.1.5.2 For use bottles, the expiration date is three years unless specifically stated in the procedure.

5.1.6 For all stock and use bottles with no expiration date, rechecks will be performed according to the Administrative Policy for Drug Chemistry Quality Assurance to ensure reagent reliability.

5.1.7 Application of Procedures on Evidence – (Specific instructions are included in the Drug Chemistry Section Technical Procedure for Preliminary Color Tests that utilize more than one reagent.).

5.1.7.1 Add 1-2 drops of the reagent to a clean spot well or a new culture tube, and ensure no significant color develops.

5.1.7.2 Add a small amount of sample to the reagent in the spot well or culture tube.

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5.1.7.3 Observe any reaction or color produced.

5.1.7.4 Record results in the FA case file if test is being performed for casework or FA Resource Manager if test is being performed for quality control purposes.

5.1.8 Marquis

5.1.8.1 This color test reacts with opiates and amphetamines, as well as some non-controlled substances, to produce colored intermediates.

5.1.8.2 Add 8-10 drops of (40 %) formaldehyde solution to 10 milliliters of concentrated sulfuric acid, with stirring.

5.1.8.3 The expiration date for this reagent shall be one month after preparation.

5.1.8.4 Lot number: Eight digit format year/month/day/Mq/initials of preparer. Example: 20101231MqXXX

5.1.8.5 QC check: Guaifenesin produces a purple color.

5.1.8.6 Results: Opiates (heroin, oxycodone) – purple
Phenethylamines (meth)amphetamine – orange/brown
MDA/MDMA – purple/black
Aspirin – slow cherry red

In the words of the following section (found in Attachment B) of the North Carolina Crime Lab protocol "Technical Procedure for Drug Chemistry Analysis":

"5.5.15 For the use of any method to be considered of value in the identification of the controlled substance, the test shall be considered positive.

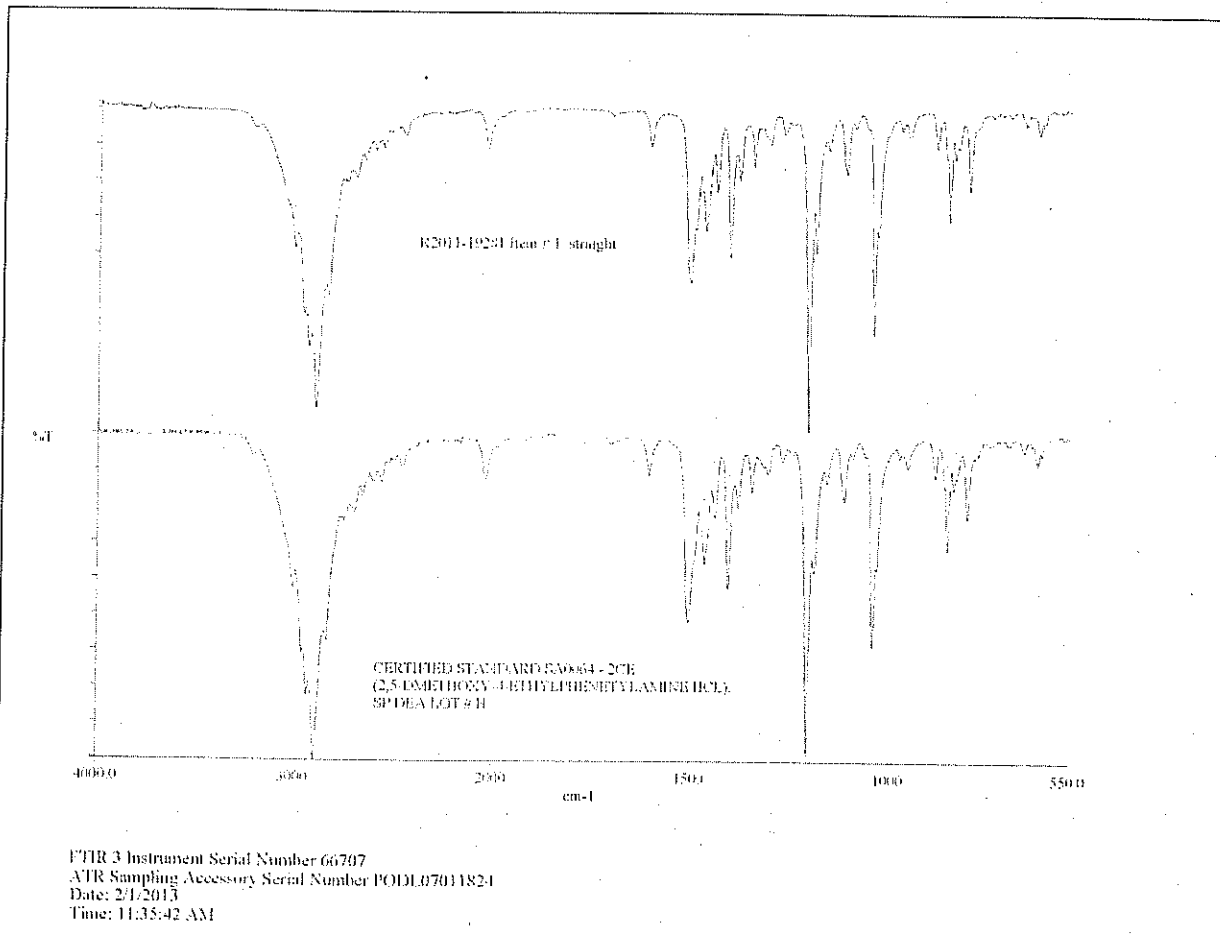
5.5.15.1 While negative tests provide useful information for ruling out the presence of a particular drug or drug class, these results have no value toward establishing the positive identification of a drug."

The Marquis test is not useful in the identification of this material. The analyst then used Fourier transform Infrared Analysis despite the further requirement that:

"5.5.12 When a Category A technique is incorporated into an analytical scheme, then at least one other technique (from either Category A, B, or C) shall be used."

The Marquis reagent test, a color test, is a Category C test which when used in this instance is of no value in the identification of a controlled substance. Therefore the analyst has only used the FTIR test, not following the requirements of the North Carolina Crime Laboratory.

The data from the FTIR analysis are presented below:



This data presents two problems. First is the weakness of the FTIR analytical tool itself. The analyst cannot know whether the peaks on the spectra are from one, two, three or a number of different materials which together mimic the spectrum of the material "identified". This situation can be seen from the passage in Attachment C by Brian Smith. This same analyst will be seen later in this report to recognize the issues with analysis of mixtures in her use of the gas chromatograph/mass spectrometer, (GC/MS). The GC separates components before analysis just as this analyst should have attempted to do before the IR analysis. She simply assumed that this material was pure, an assumption that cannot be made simply by looking at the powder presented to her.

The second problem this data presents is a legal problem. North Carolina General Statute 15A-903(d) states:

(d) Any person who willfully omits or misrepresents evidence or information required to be disclosed pursuant to subdivision (1) of subsection (a) of this section, or required to be provided to the prosecutor's office pursuant to subsection (c) of this section, shall be guilty of a Class H felony. Any person who willfully omits or misrepresents evidence or information required to be disclosed pursuant to any other provision of this section shall be guilty of a Class 1 misdemeanor.

Where subdivision (1) subsection (a) reads as follows:

§ 15A903. Disclosure of evidence by the State – Information subject to disclosure.

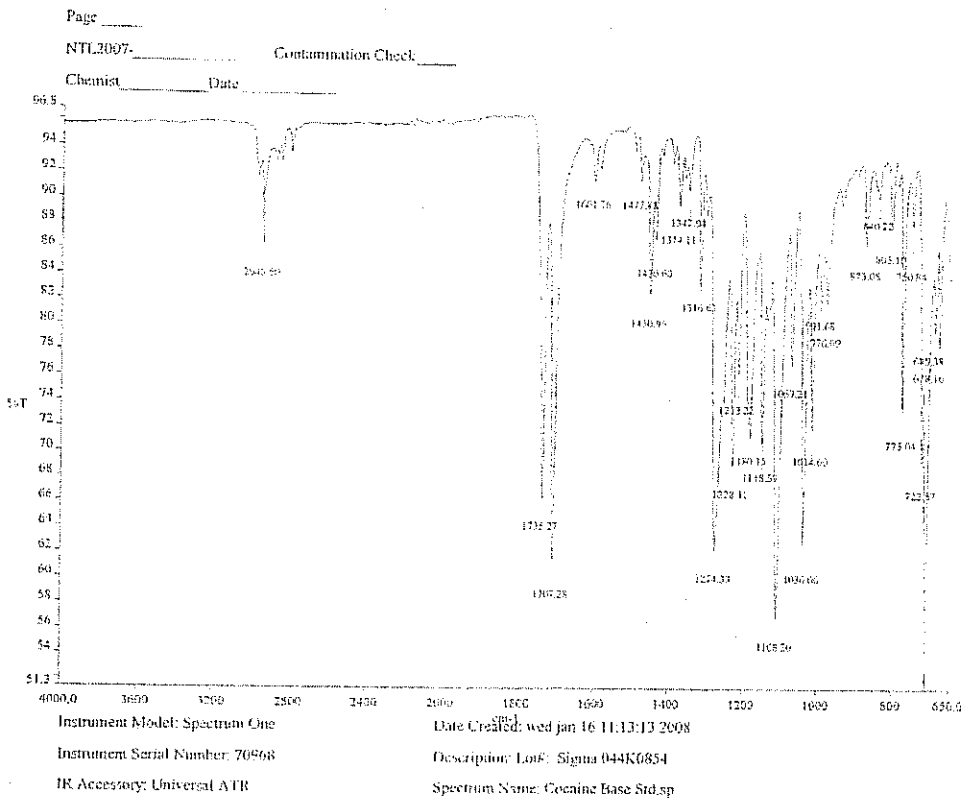
(a) Upon motion of the defendant, the court must order:

(1) The State to make available to the defendant the complete files of all law enforcement agencies, investigatory agencies, and prosecutors' offices involved in the investigation of the crimes committed or the prosecution of the defendant.

a. The term "file" includes the defendant's statements, the codefendants' statements, witness statements, investigating officers' notes, results of tests and examinations, or any other matter or evidence obtained during the investigation of the offenses alleged to have been committed by the defendant. When any matter or evidence is submitted for testing or examination, in addition to any test or examination results, all other data, calculations, or writings of any kind shall be made available to the defendant, including, but not limited to, preliminary test or screening results and bench notes.

The data (spectra) which the analyst has provided does not allow the defendant to compare data from the two spectra which are presented. The computer which is attached to the FTIR instrument itself stores the actual data which is used to present the spectra, however the resolution on the x axis, labeled "cm-1", is so fine that a visual comparison is not possible. That data is in the records of the crime lab and has been suppressed in violation of 15A-903(d).

An example of data presentation is comparison of cocaine spectra with peaks labeled is a follows:



In summary, the analyst who opined that the material was identified as 2,5 dimethoxyethylphenethylamine(2C-E) did not adhere to the protocol requirements of the North Carolina Crime Laboratory and therefore this laboratory would not stand behind this work product...according to its own protocols.

R201206840

Item #1 submitted on April 19, 2012 by Sheleathea Wright-Quick of the Chapel Hill Police Department in lab number R201206840 described as a "Brown paper bag containing plant material". The results of the examination are reported in this report as:

RESULTS OF EXAMINATION:

Item 1

Marijuana - Schedule VI.

Net weight of material - 6.68 (+/- 0.04) grams.

The data presented by the analyst indicates that the analyst observed leaves, stems, hairs but no seeds and that as a result of the color spot test, the Duquenois-Levine test, the analyst observed the "proper color" in the test solution.

The protocol presently used by the North Carolina Crime Laboratory does not identify cannabis sativa to the exclusion of most other plants. This report includes literature concerning the use of this test from its "invention" in 1938 to a recent article in the Texas Tech Law Review which explains the issue with forensic identification of cannabis sativa. To summarize the issues, the analytical protocol used by the North Carolina Crime Lab was first put into use in forensic labs in about 1938. In 1969 Dr. George Nakamura published a white paper suggesting a proper validation study to be conducted on this protocol. However Dr. Nakamura's study only included 31,874 species. There are 1,015,000 named plants in the Index Kewensis and arguments addressing synonymy among the listed plant names results in different individuals claiming the number of unique plant species ranging from about 260,000 to over 400,000 species. This number means that the forensic examiner cannot render an opinion that he has identified the material he observes to be marijuana to the exclusion of most other plants. For instance, when the analyst in this matter observed leaves, that of course could refer to any number of leaf bearing plants, hundreds of thousands of them. When the analyst observed stems, the same thing applies. When the analyst observed "hairs" a referral to Nakamura's paper shows us that even Nakamura could not differentiate 82 different species of plants microscopically among the 600 that he found had hairs on them. So the microscopic analysis does not "identify" marijuana.

The next issue is with testing with the Duquenois-Levine test. In State v. Tate, 300 N.C. 180, 265 S.E.2d 223 (N.C., 1980), the court held that the test was not scientifically accepted, accurate or reliable. The test was never meant to be more than a presumptive test for the presence of marijuana and depends upon subjective color perception, following accepted protocol very closely and understanding that very limited testing has been conducted since 1938 when the test was introduced. Duquenois and Negm, inventors of the test, did not have available to them an understanding of how the test actually functioned. As Thornton and Nakamura pointed out in their 1972 paper, the test is still somewhat enigmatic. The logic that because marijuana causes the solution to turn purple means that anything put into the solution that turns it purple is therefore marijuana is deeply flawed. Nakamura and Thornton's approach was based on an assumption that one type of material would cause the solution to turn purple and they ignored the fact that the Chemical Abstracts Registry lists millions of known chemical

compounds (now over 60,000,000 known compounds), millions of which in combination might cause the same effect. Essentially what the NC Crime Lab protocol does is to put two presumptive tests together and assume that positives from each therefore mean the presence of marijuana. The lab has never conducted its own validation studies and therefore use of the protocol by the NC Lab is in violation of its quality assurance program.

A further issue involves the use of the instrument that determines the "proper" in the Duquenois-Levine test. The defendant has not been provided with the results of color blind tests conducted on the analyst in this matter. Because the detected wavelengths of light for this test are described in the scientific literature as ranging from blue to purple, any vast number of materials might cause the same color change. And without knowing if the analyst can resolve blue from purple from red with her eyes, the defendant is denied information needed to determine the validity of her opinion.

If we apply Rules of Evidence to this analysis we see that Rule 702 sets the standard which has not been met in this case.

Rule 702. Testimony by experts.

(a) If scientific, technical or other specialized knowledge will assist the trier of fact to understand the evidence or to determine a fact in issue, a witness qualified as an expert by knowledge, skill, experience, training, or education, may testify thereto in the form of an opinion, or otherwise, if all of the following apply:

- (1) The testimony is based upon sufficient facts or data.
- (2) The testimony is the product of reliable principles and methods.
- (3) The witness has applied the principles and methods reliably to the facts of the case

The opinion that this material is cannabis sativa is not based upon sufficient facts or data. The opinion has not been shown to be the product of reliable principles and methods because the North Carolina Crime lab has not validated this protocol.

In summary, the analysis of this material using an unvalidated protocol cannot result in a scientifically sound opinion that this material is actually cannabis sativa.

R201207281

Item #1 submitted on May 01, 2012 by Sheleathea Wright-Quick of the Chapel Hill Police Department in lab number R201207281 described as a "Brown paper bag containing plant material". The results of the examination are reported in this report as:

RESULTS OF EXAMINATION:

Item I
Marijuana - Schedule VI.
Net weight of material - 6.60 (+/- 0.04) grams.

The data presented by the analyst indicates that the analyst observed leaves, stems, hairs but no seeds and that as a result of the color spot test, the Duquenois-Levine test, the analyst observed the "proper color" in the test solution.

The same analysis that was presented for Item #1 of case R201206840 above is relevant here. The North Carolina Crime Lab has not validated it's marijuana analysis protocol.

R201208315

Item #1 submitted on May 16, 2012 by Sheleathea Wright-Quick of the Chapel Hill Police Department in lab number R201208315 described as a "Paper bag containing plant material". The results of the examination are reported in this report as:

RESULTS OF EXAMINATION:

Item 1

Marijuana - Schedule VI.

Net weight of material - 102.01 (+/- 0.04) grams

The data presented by the analyst indicates that the analyst observed leaves, stems, hairs but no seeds and that as a result of the color spot test, the Duquenois-Levine test, the analyst observed the "proper color" in the test solution.

The same analysis that was presented for Item #1 of case R201206840 above is relevant here. The North Carolina Crime Lab has not validated its marijuana analysis protocol.

Item #2 submitted on May 16, 2012 by Sheleathea Wright-Quick of the Chapel Hill Police Department in lab number R201208315 described as a "Plastic bag containing:

Four clear capsules containing off-white powder material." The results of the examination are reported in this report as:

Item 2

Three capsules were individually analyzed and were each found to contain:

3,4-Methylenedioxymethcathinone - Schedule I

(A substituted 2-amino-1-phenyl-1-propanone).

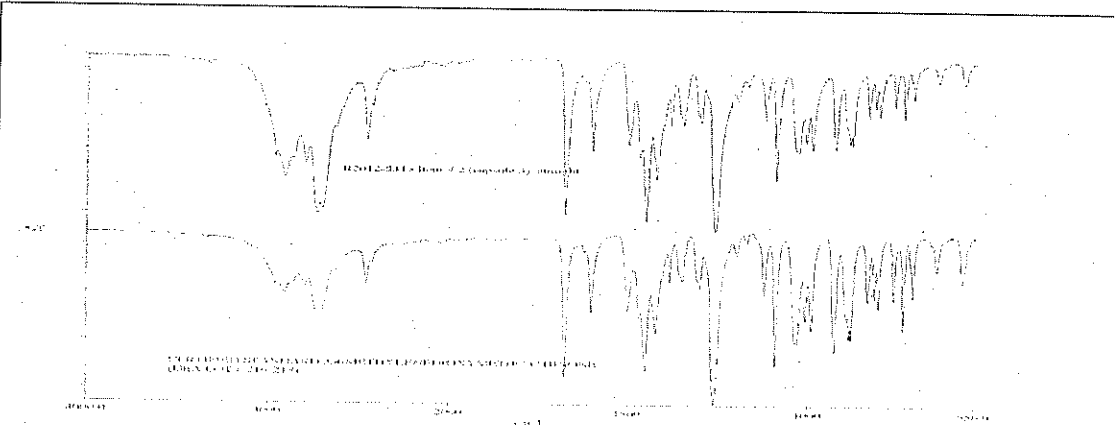
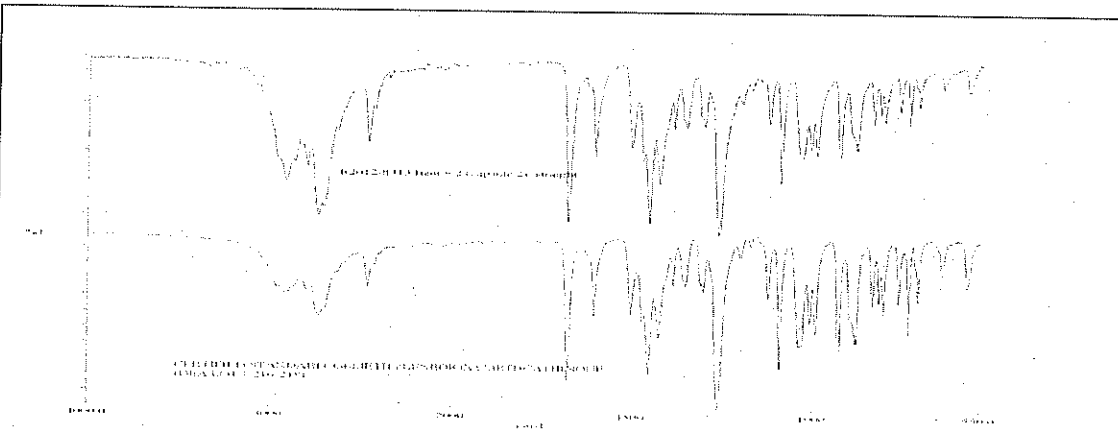
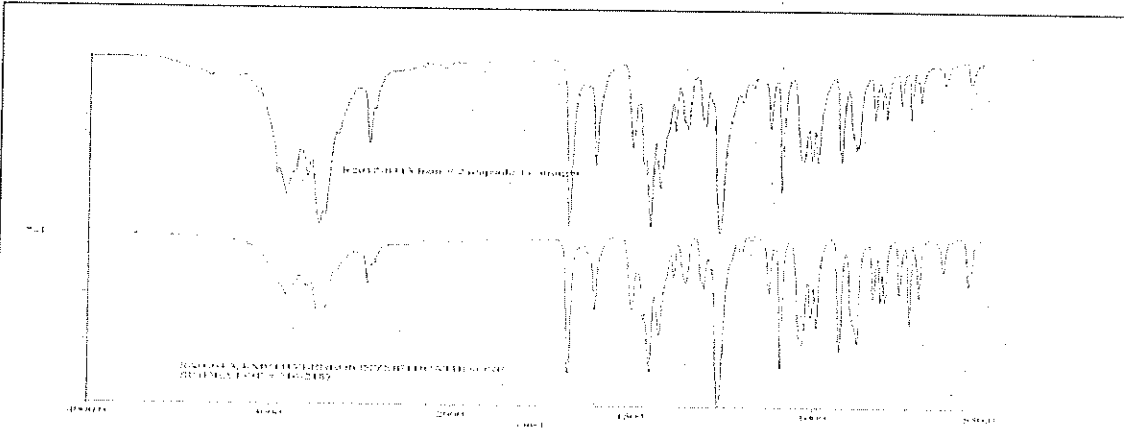
Net weight of powder material - 0.51 (+/- 0.07) gram.

One capsule - No chemical analysis.

Gross weight (capsule and powder) - 0.3 gram.

The report on the analysis of this material indicates that a Marquis reagent was first utilized with the resultant color of the solution being yellow. Then the analyst used FTIR analysis. The analysis which was presented for Item #1 R201119281 concerning the use of the Marquis reagent applies here. The Marquis reagent gives a negative response for the compounds which are listed in the NC Crime Lab protocol and therefore is not useful for determining identification of this compound.

The FTIR analysis gave the following data:



These FTIR spectra, though very much alike, have significant differences. Though the data of the standards analyzed are each labeled with "DEA Lot # 216-218", in fact the spectra of these standard samples vary among themselves. Look at the energy data in the area of 1000 cm⁻¹ to see that the presence of doublet peaks at about 800 cm⁻¹ is in the spectrum for the second and third samples but not in the first sample. The peak shapes are different in this area. The same issue arises with the peaks at about 1050 cm⁻¹. Doublets in two and three but not in one. The relative peak heights at about the 1000 cm⁻¹ area also varies significantly between the standards and the unknowns. Either the standard is not pure, the instrument is not functioning consistently between samples or the resolution at which the data is collected is not sufficient. The differences in the spectra must be explained or else the validity of this opinion must be questioned. Very small differences in spectra can mean very significant differences in structure of the molecules being analyzed.

Rule 702. Testimony by experts.

(a) If scientific, technical or other specialized knowledge will assist the trier of fact to understand the evidence or to determine a fact in issue, a witness qualified as an expert by knowledge, skill, experience, training, or education, may testify thereto in the form of an opinion, or otherwise, if all of the following apply:

- (1) The testimony is based upon sufficient facts or data.
- (2) The testimony is the product of reliable principles and methods.
- (3) The witness has applied the principles and methods reliably to the facts of the case

In the case the opinion of the analyst is not based upon sufficient facts or data. The Marquis test is negative and can not be used to support identification according to NC Crime Lab protocols. In this case FTIR is used but not used correctly. The material is analyzed as it is presented without concern about possibility that it is a pure compound or a mixture of compounds. The FTIR provides data that is not explained sufficiently. The differences in spectra are significant. In this case FTIR is applied but in the manner that it is applied one can not know the error rate thus the reliability. The total protocol, use of Marquis color test as well as FTIR, is not supported by the NC Crime Laboratory.

Because the FTIR data is not complete despite the fact that the NC Crime lab obviously collected it, there is a violation of NC GS 15A-903(d).

Item #3 submitted on May 16, 2012 by Sheleatha Wright-Quick of the Chapel Hill Police Department in lab number R201208315 described as a "Closed plastic bag containing off-white powder material". The results of the examination are reported in this report as:

Item 3

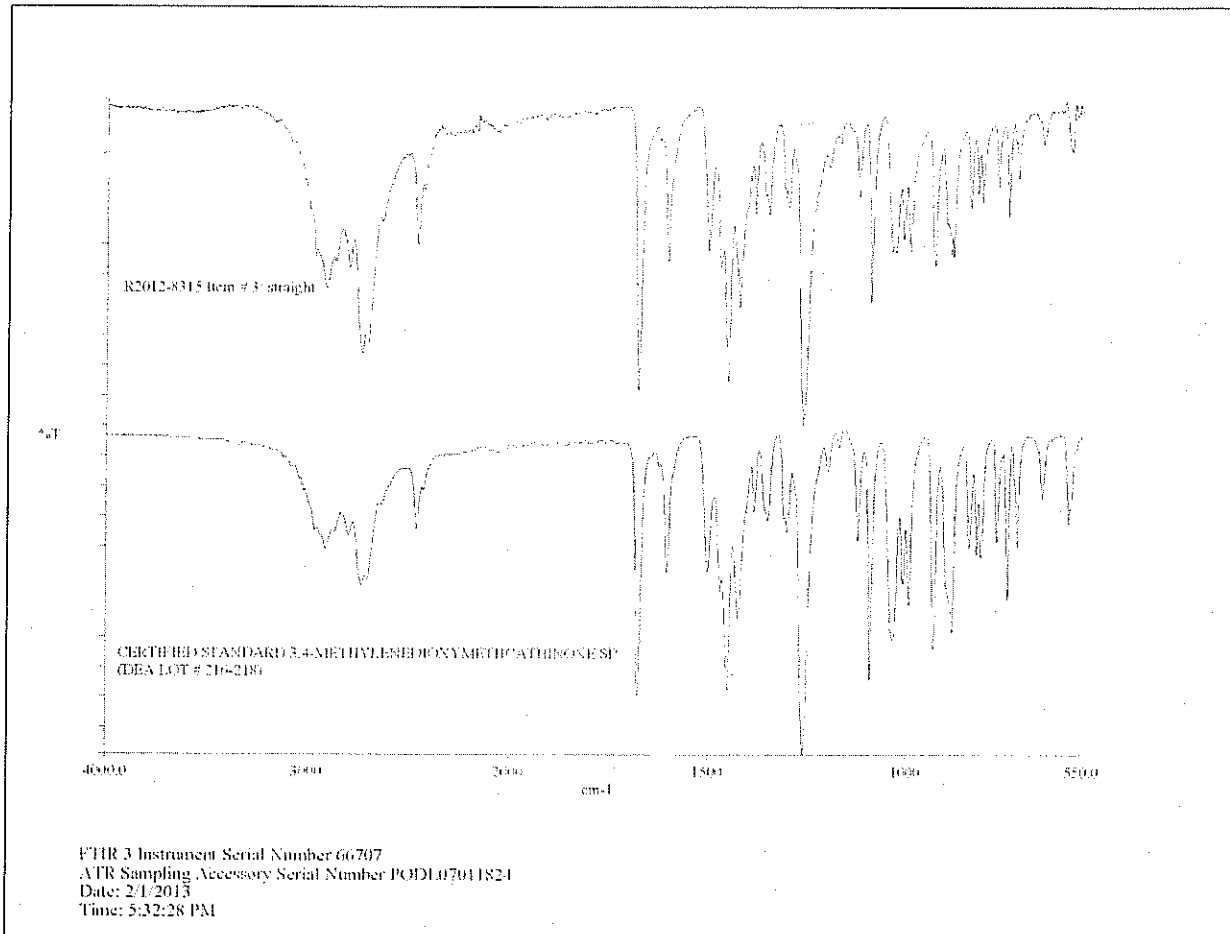
3,4-Methylenedioxymethcathinone - Schedule I

(A substituted 2-amino-1-phenyl-1-propanone).

Net weight of material - 0.93 (+/- 0.04) gram

In this analysis the analyst utilized the Marquis Reagent test, the Cobalt thiocyanate test and microcrystalline analysis. Both the Marquis Reagent test and the microcrystalline analysis tests gave negative results and, explained above, cannot be used for identification purposes. The Cobalt thiocyanate test gave a positive for the presence of cocaine. However after reviewing the FTIR data the analyst opined that the white powder was 3,4-Methylenedioxymethcathinone. The significant differences in the spectra below indicate that the FTIR data is not consistent with the presence of this

compound.



The difference in the areas about 1000 cm-1 as well as the doublet in the area about 800 cm-1 are significant both in the presence of the doublets as well as the difference in peak height ratios seen about the 1000 cm-1 area.

Because the FTIR data is not complete despite the fact that the NC Crime lab obviously collected it, there is a violation of NC GS 15A-903(d).

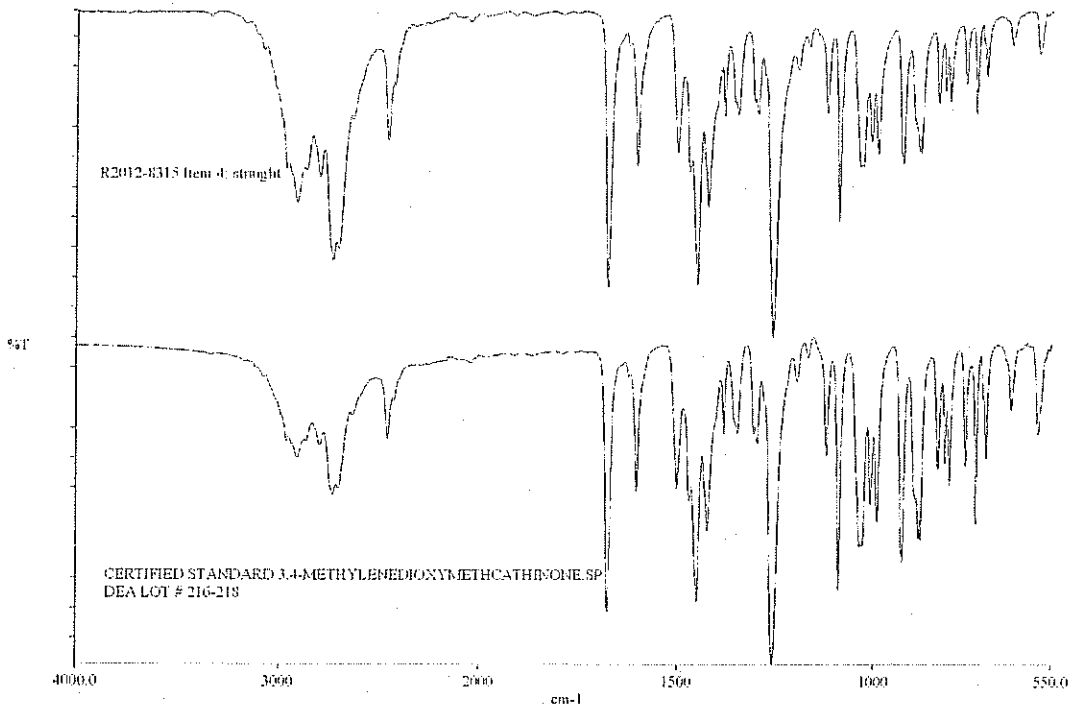
It is troubling that the analyst in this matter will change her protocol between samples analyzed seemingly without any thought and in violation of NC Crime Lab procedures. She opined the presence of the same compound in Item #2 as in Item #3 and yet did not use the same protocol. North Carolina Crime Lab Quality Assurance Protocol (See Attachment D) requires that protocols be validated. No validation studies have been presented to this reviewer at this time for any of these protocols used in this case.

Item #4 submitted on May 16, 2012 by Sheleathea Wright-Quick of the Chapel Hill Police Department in lab number R201208315 described as a "Closed and folded plastic bag containing off-white powder material". The results of the examination are reported in this report as:

Item 4

3,4-Methylenedioxyamphetaminone - Schedule I
(A substituted 2-amino-1-phenyl-1-propanone).
Net weight of material - 0.12 (+/- 0.04) gram

The analyst utilized the Marquis Reagent test, the Cobalt thiocyanate test and Microcrystalline Analysis as well as FTIR of the "straight" material. The Marquis Reagent and the Microcrystalline test gave negative results and cannot be used as a basis for identification according to North Carolina Crime lab protocols. The Cobalt thiocyanate test gave a positive for the presence of cocaine. Due to the inconsistency in the FTIR data for the DEA Lot # 216-218 standard, any opinion concerning the value of the FTIR data in this case is suspect. The spectra presented below do not suffer from the same issues as those in FTIR data from Items 2 and 3. However the inconsistency remains a piece of data which must be explained here. The spectral comparison is:



FTIR 3 Instrument Serial Number 66707
ATR Sampling Accessory Serial Number PODL07011824
Date: 2/2/2013
Time: 12:32:31 PM

The protocol utilized in this analysis to establish the identification of a chemical compound is in violation of North Carolina Crime lab protocols demanding the a Category A and Category C analytical tool be utilized as long as the Category C provided positive data. The analyst used three Category C analytical tools, none of which gave positive data for the presence of the material "identified" by the analyst here.

Item #5 (described as "sealed manila envelope reported to contain unknown powder) and Item #6 (described as Folded piece of foil containing (amodified rectangular piece of paper, divided by markings into 5 squares of consistent size (~1/4 inch X ~1/4 inch) submitted on May 16, 2012 by Sheleathea Wright-Quick of the Chapel Hill Police Department in lab number R201208315 were not analyzed according to this report.

Item #7 submitted on May 16, 2012 by Sheleathea Wright-Quick of the Chapel Hill Police Department in lab number R201208315 described as Sealed (taped, initialed and dated) brown paper bag containing tan/ brown plant material (mushrooms: caps and stems). The results of the analysis are reported as:

Item 7

Psilocin - Schedule I.

Net weight of plant material - 13.02 (+/- 0.04) grams

analyst is following measurement protocol -

The material was analyzed utilizing a chemical spot test based upon paradimethylaminobenzaldehyde (PDMAB) which gave results of a purple color and gas chromatography/mass spectrometry. The use of PDMAB for the identification of psilocin is according to the North Carolina Crime Lab protocols and the formation of the purple solution is a positive indicator for the presence of psilocin. The GC/MS data is consistent with the presence of psilocin. The North Carolina Crime Lab's requirement that a Category C and a Category A method be used with positive results in order for an analyst to identify a compound is in this analytical scheme joined by a Category B method (Gas chromatography) resulting in the identification of psilocin.

The analysis of item 7 conducted in a proper manner raises the question as to why the analyst chose in this analysis to follow proper protocol and in other analyses in this matter chose to ignore the North Carolina Crime Lab's protocols.