

Hamline University
Department of Criminal Justice and Forensic Science

**CJFS 3425: Forensic Chemistry
Lab Manual**

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CJFS 3460: Laboratory Notebook & Report

Each student is required to keep a laboratory notebook to record observations associated with laboratory exercises. The book must be bound so that the pages are not removable. Composition “marble” notebooks (approximately $7\frac{1}{2}$ ” x 10”) with sewn bindings are ideal. It is best to select a notebook that has a wide (approximately one inch) top and left margin on each page. The cover of the notebook must indicate the **student’s name, course number and semester/year**. Entries must be made in either black or blue water-insoluble ink. The first page of the notebook must be reserved for the table of contents. A table of contents “template” is provided in Appendix A. You must neatly cut and tape this template into your notebook for use throughout the semester. An example of usage is shown below. Please ensure that each column of the table of contents is complete before you hand in an exercise for grading.

Table of Contents Entry Example

Exercise	Pages	Transferred By	Received By	Transferred By	Received By	Grade
1. Microscopy	2-10	Student's Initials 01-Jan-2021	JSS 01-Jan-2021	JSS 07-Jan-2021	Student's Initials 07-Jan-2021	0.95

Each page of the notebook must be consecutively numbered in the top margin (outer edge of each page furthest from the binding). This must be completed for all pages in the notebook before the second class meeting. Just before commencing a lab exercise, the student must sign in by placing the date in the left margin of the page, and writing “*I began lab work at XXXX hours.*” An example is shown in appendix B. Line out any remaining unused space on this line and initial the entry. When you leave the lab, repeat the process, but indicating the time you exited the lab.

The top line of each page should include the date on which entries on that page were made. When more than one page is used for entries for the same exercise on a particular day, all subsequent pages should indicate the date followed by the word “continued” in parentheses, followed by a time stamp of “XXXX hours”. *Be sure to record your activities in a manner that would withstand scrutiny by any outside observer. Your reputation depends on your honesty and integrity.*

Data and observations recorded for each exercise must be entered into the notebook contiguously. You may not leave blank spaces in your notes for the purpose of “filling in” information at a later date or time. Please record all observations in your notebook (for knowns, controls and unknowns) at the time they are made and avoid summarizing data. If you repeat a number of tests, the notebook should normally reflect multiple entries. Always document in the present or past tense and only document what you did (*e.g.*, not what you intend to do).

If you make an error in your notebook, you can correct it by placing a single line through the entry and placing the corrected entry to the right of the error. Always initial and date each correction. If a correction is lengthy or found after including other observations, mark the error using the date, initials, “ * pp. ” and the page number of the next available blank line in your notebook; write the correction on this line under an underlined heading such as: * Correction of entry made on page #. Do not attempt to obliterate an entry either by inking it out or using whiteout. Never overwrite data.

When inserting a chart, sketch attachment, or other loose paper into your notebook, tape down one side onto a blank page (applying tape to the back and front side of the edge). If necessary, fold the insert so that it is not protruding from your notebook. In the middle of the blank space beneath the taped in material, indicate what the page has been reserved for. Finally, draw a diagonal line through the blank space beneath the taped in material (breaking intermittently so as to avoid crossing out writing that denotes what the page is reserved for). Similarly, a diagonal line should be drawn through any unused

portion of any page. All diagonal lines should be initialed and dated. If you only fill half a line in the notebook, be sure to use a straight solid line to line out any remaining space.

The required date format is day-month-year (for example, 01-Jan-2021). When the date is comprised of a single digit, use a preceding zero. Months are designated by the first three letters (for example, Sep, Oct, Nov). The date format is not to have any spaces between the day, month, and year. The required time format is military time (*e.g.*, 6:15 PM is expressed 1815 hours).

The last entry in the notebook for each exercise with unknowns should be a brief conclusion regarding the results. Make sure that you are signed into the notebook, then start a new page, and label the first line (after the margin) with the following "Report of Evidence XX:" where "XX" refers to the unknown number. State your conclusions, and then sign, date and time stamp the report. An example is provided in appendix B. If the exercise is accompanied by a typed summary, the notebook report page should include a statement that directs the reader to the typed summary report (including specifics regarding the author and title of the report, as well as the total number of pages included in the typed document). Be sure to include the page number of the "report page" in the table of contents. When you are finished, hand the notebook in to the instructor or teaching assistance for grading. To do this, use the chain of custody columns that are included in the table of contents page of your notebook. If the exercise does not involve an unknown, a formal report is still required; simply make a statement that you have completed the exercise (*e.g.*, "Formal Completion of Exercise YY:"), where "YY" is the laboratory activity. Provide some type of summary statement regarding the completed activities and then sign, date and time stamp the statement.

As a final note on documentation, students must learn to document relevant information in an efficient manner. Devise protocols and refer to the protocol each time instead of tediously rewriting information that is available in an earlier portion of your notebook. A lawyer, judge, or other scientist may scrutinize the notebook of a forensic scientist. Put yourself in the position of the opposing team and think about how your documentation may be attacked.

Handling of Unknowns

Please treat unknown samples as if they were evidence. Each student must have direct control over his or her evidence at all times. Before a student subjects an unknown material to a particular type of analysis, he or she must demonstrate that they have "mastered" the technique on known exemplars first; never waste unknown material by performing a test you are uncertain about on a questioned sample. In rare instances in the classroom, your instructor may override this rule. This decision is not taken lightly, but at times will be the best use of instructional hours and resources. Of course, the reasoning behind such a decision does not exist when dealing with actual evidence, so you should not circumvent this rule outside of pre-specified instances in this class.

When obtaining an unknown, you will be required to fill out a chain of custody form. Four copies are provided to you at the end of this document in appendix A. The chain of custody form will be returned to the instructor who will hold it until the student returns the evidence. Whenever reasonable, unknowns will be issued in sealed packages. The seals will be initialed and dated by the person who packaged the unknown. If the package is not sealed properly, seal it and document it in your notebook. Write your initials and the date over the seal.

As soon as you receive the unknown, immediately initial and date the package, and write, "*received*". Document this act in your notebook, including the location where you added permanent information to the packaging. If a lab exercise extends beyond one period or if you leave the lab, you must be properly

secure and document the storage of the unknown (you must indicate where the item is being stored).

When ready, open the packaging using a sharp instrument. Choose a location to open the package that is sufficiently distant from the original seal and note the location in your notebook. If at all possible, never break an existing seal. Near the opened area, initial and date the package and write, “*opened*”. Document this in your notebook. Make a large enough opening so that when removing the item no evidence is lost. Remove the item onto a clean piece of evidence or butcher paper to minimize contamination. Retain the original packaging. It is not necessary to reseal the evidence in its original packaging at the end of each laboratory session during the testing of that item. You must, however, properly secure the unknown, for instance, stored in some alternative manner in a secure location, when it is not in your immediate possession.

Please note that as soon as an analyst opens evidence, he or she should generally examine the item with a stereomicroscope before proceeding with any other type of testing. During this inspection, be sure to record the magnification you are using, and if appropriate, sketch important details regarding the unknown.

Once testing is complete, reseal the evidence in the packaging. Initial and date over the new seal ensuring that your notations (script) span both the packaging and the tape. Finally, write the word “*sealed*”. Document this in your notebook. Return the package to the lab instructor and document the transaction in your notebook. Complete the chain of custody form that will be given back to you. Document this activity, and **IMMEDIATELY tape the completed chain of custody form into your notebook using the appropriate taping technique.**

Notebook Grading

Each assignment and laboratory exercise will be graded based on (a.) the thoroughness of the scene investigation, (b.) notebook documentation, (c.) a typed *Criminal Investigation* report, and/or (d.) a combination of items (a.)-(c.). Keep in mind that the course instructor can demand to see and assess the notebook at any point in time, regardless of whether or not a summary report has been provided by the student. In terms of notebook documentation, some common errors are listed below. Students will be penalized for not adhering to the guidelines, and for repeatedly making the same mistake. Most errors will receive a deduction of **2% per occurrence**. As the semester progresses, if a student continues to repeat errors or commit new errors, the deduction/penalty for not progressing with your documentation skills will increase above 2% per occurrence. Please be warned that egregious errors will receive much higher deductions, regardless of when committed.

Errors	
Improper format (e.g., date, time, page #)	Incontiguous entry (some are minor, others are egregious)
Improper error correction	Improper insertion
Chain of custody omission	Improper documentation of unknown
Improper handling of unknown packaging	Insufficient testing of knowns and controls
Omission of observations or data	Naming the wrong person that issued/received an unknown
Handing in a lab late; see syllabus	Extensive summarization of observations

Help

In many cases an exercise will not contain rote instructions. Instead, the student is expected to interpret the objectives provided in the exercise, and then determine the most appropriate manner in which to proceed. It is expected that the student will consult appropriate resources prior to beginning the exercise. If the student continues to have questions or problems, he or she should consult the instructor to ask questions in-person or via email.

Appendix A. Table of Contents and Chain of Custody Templates

Please cut out and tape the above table of contents template onto the first page of your laboratory notebook. Hand-transcribed replicas of the table of contents are not permitted.

Chain of Custody Forms

Evidence ID# _____

Brief Description of Item _____

Transferred By _____ Date Transferred _____

Time Transferred _____ Location Transferred _____

Signature of Recipient _____

Transferred By _____ Date Transferred _____

Time Transferred _____ Location Transferred _____

Signature of Recipient _____

Condition of Package _____

Evidence ID# _____

Brief Description of Item _____

Transferred By _____ Date Transferred _____

Time Transferred _____ Location Transferred _____

Signature of Recipient _____

Transferred By _____ Date Transferred _____

Time Transferred _____ Location Transferred _____

Signature of Recipient _____

Condition of Package _____

Appendix B. Documentation Examples

Please follow the examples below for executing the proper documentation for the course and forensic practice in general. Additional examples will be provided on Canvas.

Signing Into Notebook

Receipt of Unknown Sample

	8
01-Jan-2021	(continued) 1030 hours
	<i>upon inspection, two prominent paper fibers protruded from the serrated edge.</i>
	<i>On this date, in room 205 (Drew Science Center), at 1033 hours, I received</i>
	<i>an unknown from Dr. Spaulding. ——————</i>
	<i>01-Jan-2021 —————— Unknown #I-13 —————— 1034 hours —————— JSS</i>
	<i>Unknown #I-13 was received from Dr. Spaulding at 1034 hours. Packaging</i>
	<i>consisted of a small coin envelope. I immediately marked the envelope with</i>
	<i>“MS 01-Jan-2021 Received” in the top left corner above “I-13”. A</i>
	<i>chain of custody form was filled out and returned to Dr. Spaulding. The</i>
	<i>seals on the unknown packaging appear intact. The seals are marked with...</i>

Unknown Report

	15
01-Jan-2021	(continued) 1420 hours
	<u>Report of Evidence I-13:</u> _____
	Unknown I-13 is consistent with exemplar "A". _____
	01-Jan-2021 _____ Mary Smith _____ 1422 hours
	Note: The report and signature formerly finish the lab. No additional information, conclusions, etc.
	can be added after the signature. Please leave the remaining space on this page blank; the instructor
	will use it for grading purposes.
	Also, keep in mind that you should not begin a report unless you have sufficient time to finish it
	(e.g., you should not take a break while writing a report; all work must be contiguous).

Please note that your notebook will be treated as evidence. You must comply with all documentation guidelines and ensure that your work meets both professional and academic standards of ethics for the forensic reporting of laboratory and field data. Dishonest entries and practices are deemed a violation of professional ethics and the student code of conduct.

Formal Completion Report

In instances where there is not an unknown sample, a formal completion for the lab exercise may be requested at the discretion of the instructor.

	15
01-Jan-2021	(continued) 1030 hours
	<u>Formal Completion of Lab 1</u>
	<i>In this lab we examined fiber, glass, and tape evidence. This experience has taught me....</i>
	01-Jan-2021 ————— Mary Smith ————— 1422 hours
	Note: The report and signature formerly finish the lab. No additional information, conclusions, etc.
	can be added. Please leave the remaining space on this page blank; the instructor will use it for
	grading purposes.
	Also, keep in mind that you should not begin a report unless you have sufficient time to finish it
	(e.g., you should not take a break while writing a report; all work must be contiguous).

REPORT OF EXAMINATION FOR EVIDENCE SUBMITTED

Recipient:
Dr. Jamie Spaulding
Director of Forensic Science Programs
219W Giddens Learning Center
Saint Paul, MN 55104-1284

Date: **January XX, 20XX**
Case ID No.: **XX-0000001**

Date Samples Received: **January XX, 20XX**

From: **George Washington; John Adams; Thomas Jefferson**
Hamline University Forensic Crime Laboratory

1. The following items were examined in the GLASS subsection of the TRACE EVIDENCE EXAMINATION SECTION:

1.1. ITEM SUBMITTED FOR ANALYSIS

K1-1 – Fragment 1 collected from the window frame of the door to residence
K1-2 – Fragment 2 collected from the window frame of the door to residence
K1-3 – Fragment 3 collected from the window frame of the door to residence
K1-4 – Fragment 4 collected from the window frame of the door to residence
K1-5 – Fragment 5 collected from the window frame of the door to residence
K1-6 – Fragment 6 collected from the window frame of the door to residence
K1-7 – Fragment 7 collected from the window frame of the door to residence
K1-8 – Fragment 8 collected from the window frame of the door to residence
K1-9 – Fragment 9 collected from the window frame of the door to residence
K1-10 – Fragment 10 collected from the window frame of the door to residence
Q1-1 – Glass sample collected from the submitted right glove collected from Jim Anderson
Q1-2 – Glass sample collected from the submitted right glove collected from Jim Anderson
Q1-3 – Glass sample collected from the submitted right glove collected from Jim Anderson

1.2. RESULTS OF EXAMINATION:

K1 consists of ten samples collected from the broken window of the door at the residence of the incident. The ten samples of K1 were sampled from different areas of the already-broken window and compared to the submitted questioned samples. The submitted question samples were collected from the right glove of the pair submitted from Jim Anderson.

Based on the examinations conducted Q1-1, Q1-2, and Q1-3 could not be differentiated from K1. The questioned glass samples showed no differences in refractive index as all measured refractive indices fell within the measured range of K1 (Appendix 1, 2). Through further fully quantitative instrumental analysis, no differences were found in the elemental composition of the known and questioned glass samples (Appendix 3, 4, 5, 6). Instrumental analysis was conducted by measuring sixteen separate elemental concentrations within each sample. These concentrations were compared between the known sample and all questioned samples (Appendix 7). Accordingly, all questioned samples (Q1-1, Q1-2, and Q1-3) originated from the same source (window pane from

door of residence) represented by K1 or from another broken window that was manufactured at the same plant at approximately the same time period (*Level II Association*). This level of association was reached because the likelihood of observing two samples of broken glass sharing matching concentrations for sixteen different elements as a result of random chance, is expected to be very low if the samples are indeed from different sources.

The following techniques were utilized in the examination of these items: visual and microscopic observations, physical measurements, refractive index using a microscope – glass refractive index measurement (GRIM) workstation, and elemental analysis with laser ablation – inductively coupled plasma mass spectrometry (LA-ICPMS). Please reference the *Appendix* for data collected during the analysis utilizing the above techniques.

1.3. FURTHER ANALYSIS REQUESTED:

None at this time.

2. INTERPRETATION:

The following descriptions are meant to provide context to the levels of opinion reached in this report. Every type of conclusion may not be applicable in every case nor for every material type.

Level I Association: A physical match; items physically fit back to one another, indicating that the items were once from the same source.

Level II Association: An association in which items are consistent in observed and measured physical properties and/or chemical composition and share atypical characteristic(s) that would not be expected to be readily available in the population of this evidence class.

Level III Association: An association in which items are consistent in observed and measured physical properties and/or chemical composition and, therefore, could have originated from the same source. Because other items have been manufactured that would also be indistinguishable from the submitted evidence, and individual source cannot be determined.

Level IV Association: An association in which items are consistent in observed and measured physical properties and/or chemical composition and, therefore, could have originated from the same source. As compared to a *Level III Association*, items categorized with a *Level IV* share characteristics that are more common amongst these kinds of manufactured products. Alternatively, an association between items would be categorized as a *Level IV* if a limited analysis was performed due to characteristics or size of the specimen(s).

Level V Association: An association in which items are consistent in some, but not all, physical properties and/or chemical composition. Some minor variation(s) exist between the known and questioned items and could be due to factors such as heterogeneity, contamination of the sample(s), or having a sample of insufficient size to adequately assess homogeneity of the entity from which it was derived.

Inconclusive: No conclusion could be reached regarding an association/elimination between the items.

Elimination: The items were dissimilar in physical properties and/or chemical composition, indicating that they did not originate from the same source.

3. REMARKS:

The supporting documentation for the opinions and interpretations expressed in this report are retained in the Hamline University Forensic Crime Laboratory Case Archives.

For questions regarding the status of remaining forensic examinations, please contact Hamline University Forensic Crime Laboratory Request Coordinator Dr. John Smith.

For questions pertaining to the content of the report please contact either George Washington; John Adams; Thomas Jefferson.

George Washington

George Washington
Trace Evidence Examination Section
Hamline University Forensic Crime Laboratory

John Adams

John Adams
Trace Evidence Examination Section
Hamline University Forensic Crime Laboratory

Thomas Jefferson

Thomas Jefferson
Trace Evidence Examination Section
Hamline University Forensic Crime Laboratory

This report contains the opinions/interpretations of the examiner(s) who issued the report.

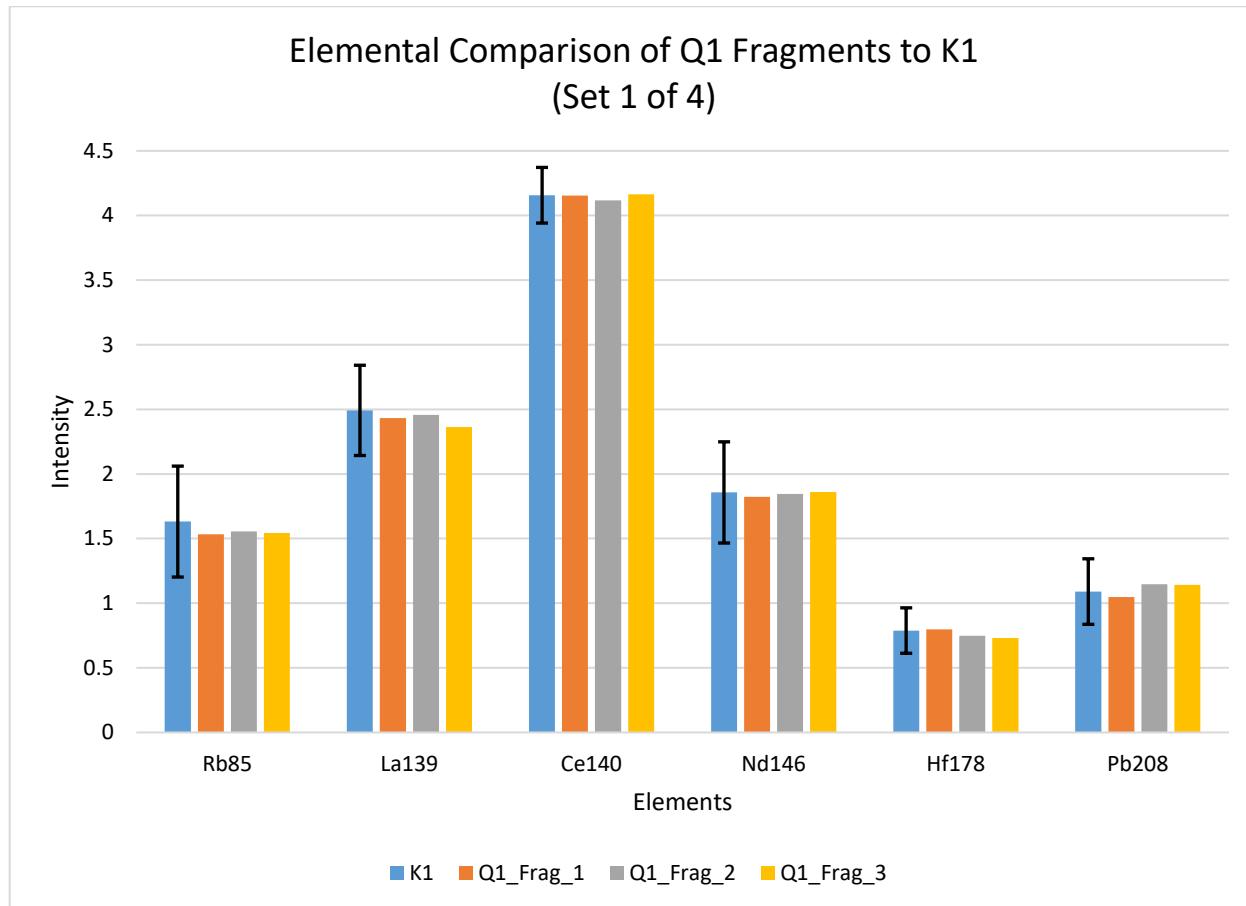
4. APPENDIX

	K1	Q1 1	Q1-2	Q1-3
Measurement 1	1.51870	1.51868	1.51869	1.51865
Measurement 2	1.51872	1.51870	1.51871	1.51870
Measurement 3	1.51868	1.51873	1.51870	1.51873
Measurement 4	1.51870	1.51867	1.51868	1.51867
Measurement 5	1.51867	1.51881	1.51870	1.51870
Measurement 6	1.51871	--	--	--
Measurement 7	1.51872	--	--	--
Measurement 8	1.52874	--	--	--
Measurement 9	1.51865	--	--	--
Mean	1.51981	1.51872	1.51870	1.51869
Standard Deviation	3.359E-3	5.6303E-5	1.1402E-5	3.0822E-5
Relative Standard Deviation	0.22 %	0.0037 %	0.00075 %	0.0020 %

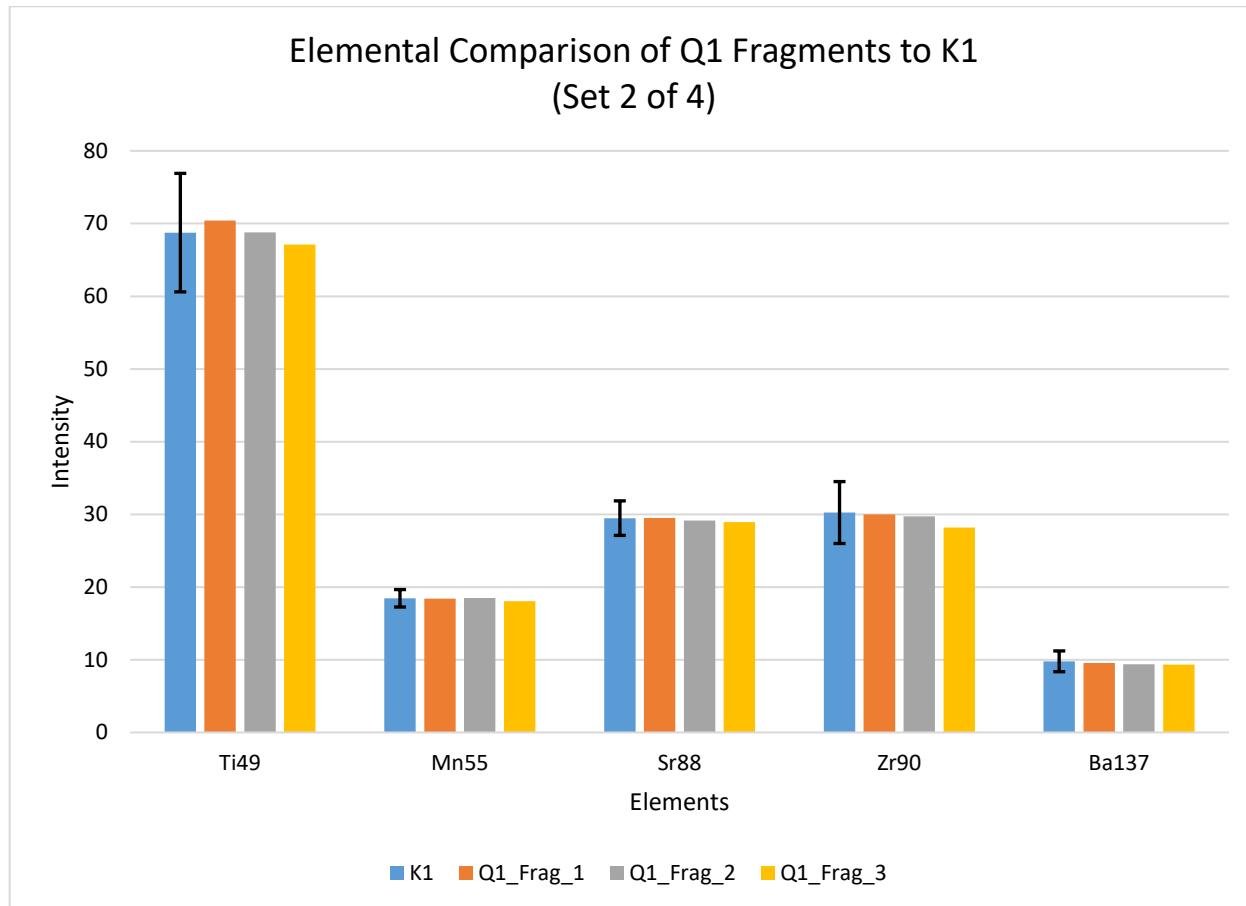
Appendix 1: Refractive Index Measurements for Glass K1 and Q1. The table contains the refractive index test results of known fragment samples comprising K1 and questioned samples Q1.

	K1	Q1-1	Q1-2	Q1-3
Mean	1.51981	1.518718	1.518696	1.51869
Max Value	1.52874	1.51881	1.51871	1.51873
Min Value	1.51865	1.51867	1.51868	1.51865
Range	1.51865-1.52874	1.51867-1.51881	1.51868-1.51871	1.51865-1.51873
Comparison to K1	--	Indistinguishable	Indistinguishable	Indistinguishable

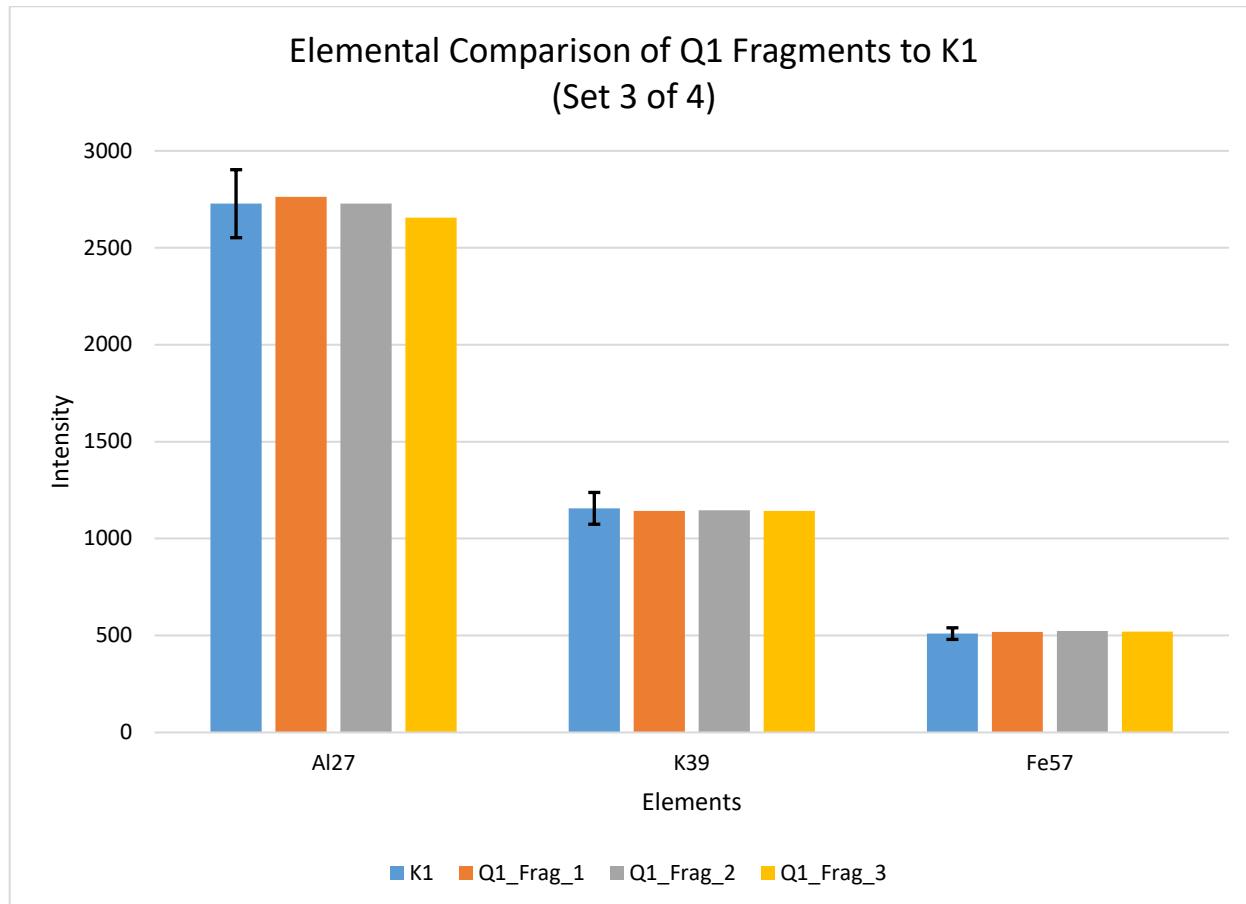
Appendix 2: Summary of Refractive Index Comparison Results for Glass K1 and Q1. The table shows the comparison of K1 to Q1 in terms of refractive index. Shown at bottom is whether the questioned samples can be distinguished from K1 by refractive index.



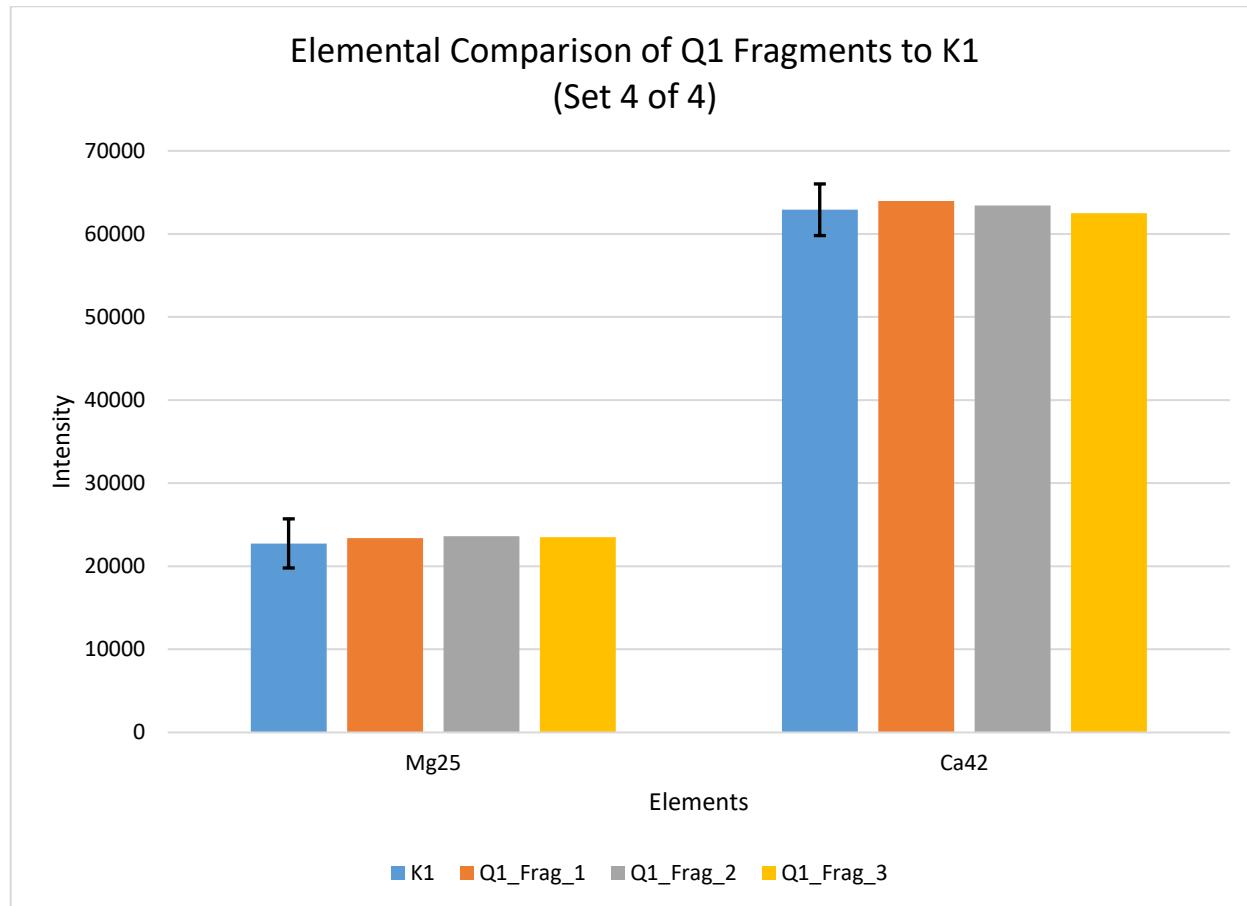
Appendix 3: Visual Summary of Rb, La, Ce, Nd, Hf, and Pb Profiling Results for Glass K1 and Q1. Shown is the average measurement of each element taken for Q1 by LA-ICPMS, selected for ease of viewing on the same chart. The lower and upper bounds of K1 represent four standard deviations below and above the average of the K1 measurement, respectively. The Q1 samples fall within this range meaning they are indistinguishable from K1 for each element. Please see Appendix 28, 29, and 30 for the remaining ten elements compared.



Appendix 4: Visual Summary of Ti, Mn, Sr, Zr, and Ba Profiling Results for Glass K1 and Q1. Shown is the average measurement of each element taken for Q1 by LA-ICPMS, selected for ease of viewing on the same chart. The lower and upper bounds of K1 represent four standard deviations below and above the average of the K1 measurement, respectively. The Q1 samples fall within this range meaning they are indistinguishable from K1 for each element. Please see Appendix 29 and 30 for the remaining five elements compared.



Appendix 5: Visual Summary of Al, K, and Fe Profiling Results for Glass K1 and Q1.
Shown is the average measurement of each element taken for Q1 by LA-ICPMS, selected for ease of viewing on the same chart. The lower and upper bounds of K1 represent four standard deviations below and above the average of the K1 measurement, respectively. The Q1 samples fall within this range meaning they are indistinguishable from K1 for each element. Please see Appendix 30 for the remaining two elements compared.



Appendix 6: Visual Summary of Mg and Ca Profiling Results for Glass K1 and Q1. Shown is the average measurement of each element taken for Q1 by LA-ICPMS, selected for ease of viewing on the same chart. The lower and upper bounds on K1 represent four standard deviations below and above the average of the K1 measurement, respectively. The Q1 samples fall within this range meaning they are indistinguishable from K1 for each element.

ELEMENT	Mg25	Al27	K39	Ca42	Ti49	Mn55	Fe57	Rb85	Sr88	Zr90	Ba137	La139	Ce140	Nd146	Hf178	Pb208
K1 vs Q1 fragm1																
AVE K1	22753	2728	1156	62914	68.8	18.5	510	1.63	29.5	30.3	9.78	2.49	4.16	1.86	0.79	1.09
STDEV K1	739	44	21	777	2.0	0.3	7	0.11	0.6	1.1	0.36	0.09	0.05	0.10	0.04	0.06
K1 -4s	19796	2552	1074	59806	60.6	17.3	480	1.20	27.1	26.0	8.35	2.14	3.94	1.47	0.61	0.84
K1 +4s	25710	2903	1238	66021	76.9	19.7	540	2.06	31.9	34.5	11.21	2.84	4.37	2.25	0.96	1.34
AVE Q1	23407	2763	1143	63965	70.4	18.4	518	1.53	29.5	30.0	9.54	2.43	4.15	1.82	0.80	1.05
Significant difference?	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN
K1 vs Q1 fragm2																
AVE K1	22753	2728	1156	62914	68.8	18.5	510	1.63	29.5	30.3	9.78	2.49	4.16	1.86	0.79	1.09
STDEV K1	739	44	21	777	2.0	0.3	7	0.11	0.6	1.1	0.36	0.09	0.05	0.10	0.04	0.06
K1 -4s	19796	2552	1074	59806	60.6	17.3	480	1.20	27.1	26.0	8.35	2.14	3.94	1.47	0.61	0.84
K1 +4s	25710	2903	1238	66021	76.9	19.7	540	2.06	31.9	34.5	11.21	2.84	4.37	2.25	0.96	1.34
AVE Q1	23610	2728	1146	63411	68.8	18.5	523	1.56	29.1	29.7	9.36	2.46	4.12	1.85	0.75	1.15
Significant difference?	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN

ELEMENT	Mg25	Al27	K39	Ca42	Ti49	Mn55	Fe57	Rb85	Sr88	Zr90	Ba137	La139	Ce140	Nd146	Hf178	Pb208
K1 vs Q1 fragm3																
AVE K1	22753	2728	1156	62914	68.8	18.5	510	1.63	29.5	30.3	9.78	2.49	4.16	1.86	0.79	1.09
STDEV K1	739	44	21	777	2.0	0.3	7	0.11	0.6	1.1	0.36	0.09	0.05	0.10	0.04	0.06
K1 -4s	19796	2552	1074	59806	60.6	17.3	480	1.20	27.1	26.0	8.35	2.14	3.94	1.47	0.61	0.84
K1 +4s	25710	2903	1238	66021	76.9	19.7	540	2.06	31.9	34.5	11.21	2.84	4.37	2.25	0.96	1.34
AVE Q1	23518	2656	1143	62504	67.1	18.0	520	1.54	28.9	28.2	9.31	2.36	4.16	1.86	0.73	1.14
Significant difference?	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN

Appendix 7: LA-ICPMS Data of Comparison Between Glass K1 and Q1 Samples. Shown in the tables are the same comparisons which were visually depicted in Appendix 27, 28, 29, and 30, for each element measured by LA-ICPMS. No significant differences were seen for each element of each sample. Seen in the table is IN for each element throughout, representative of a conclusion of an inclusion as opposed to a discrimination. This indicates Q1 cannot be distinguished from K1.

Forensic Laboratory Protocols and Rules

The following are “house rules” for the usage of microscopes and other instrumentation throughout the course.

1. All microscopes should be protected by a dust cover when not in use. When removing a dust cover, please be careful not to pull the oculars/eyepieces from the head of the scope.
2. Ensure that the voltage setting for the microscope is set to a minimum before turning it on (and off). Only after turning on the scope should you increase the transformer’s voltage for comfortable illumination intensity.
3. To change magnification, use the knurled ring on the nosepiece to rotate between objectives. In other words, do not grab the barrel of an objective to rotate the nosepiece.
4. Always begin focusing with a low powered objective (highest working distance). Note that objectives are parfocal, so as you increase magnification, you will only need to make fine focus adjustments.
5. Be sure to adjust the intraocular distance (the distance between the right and left ocular) as necessary for personal preference. Most scopes in DSC-205 also have a diopter adjustment (usually located on the left ocular) to adjust for diopter differences between your right and left eye. Finally, the image of the ocular micrometer (located in the right ocular) can be adjusted to ensure that it is parfocal with the specimen; please see your laboratory instructor if you need assistance.
6. If you need to increase contrast, close down the aperture diaphragm (located on the substage condenser). If you need to increase resolution, match the numerical aperture of the objective with the numerical aperture of the aperture diaphragm.
7. When finished observing your specimen and/or performing analytical work, rotate to a low powered objective (high working distance) before removing the specimen from the stage (to avoid colliding the specimen with the objective).
8. Wipe down plastic and metal (ONLY) parts of the microscope with a KimWipe moistened with water (stage, fine and coarse focus/adjustment knobs, knurled ring on nosepiece, voltage dial, on/off switch, barrel of oculars, etc.) as necessary to maintain cleanliness.
 - (a) If you are working with biological fluids, please wipe these parts with a water:bleach mixture.
 - (b) **Note: Kimwipes cannot be used to clean optical elements on a microscope; in other words, never touch any lens on the microscope with a Kimwipe.**
 - (c) If you believe you have contaminated a lens, please consult with the laboratory instructor for cleaning/maintenance.

- (d) Please do not clean lenses by yourself unless you have been trained by your laboratory instructor regarding proper technique.
- 9. Do not forget to decrease the voltage on the scope, turn off the power, and cover the instrument with a dust cover before ending laboratory work.
- 10. The bench working area can be covered with butcher paper if needed during the class. The working area shall be left clean at the end of the class.
- 11. Used glassware (cover slides, disposable pipettes) and any broken glass shall be disposed in the glass disposable container located in the classroom.
- 12. Any sharp objects (e.g. blades) shall be disposed in the containers assigned for sharp materials.
- 13. Other analytical instrumentation shall be used as per your instructor's recommendations. Students will be allowed to use analytical instrumentation only under supervision of your instructor.
- 14. Student shall wear lab-coats and eye protection when completing lab exercises.

Lab 1: Microscopy

Objective of Lab Exercise

This lab will familiarize you with the usage of a microscope. Visual, microscopical, optical, and physical measurements of evidence are common first steps in an analytical workflow.

Lab Instructions

Part I: Micro-manipulation

Using a tungsten carbide (WC) scribe, reproduce one parking slide model from the left column of Figure 1. Start by placing the number “9” (using a marker) in the left upper most column of a clean slide. Flip the slide over so that the “9” appears as a “P”. This will be used for orientation purposes. Using the WC scribe, generate appropriate parking spaces. Each line should be made with gentle pressure and not easily visible to the unaided eye. Use a ruler as a straight edge. Scribe the numbers for each parking spot carefully. “Tape” the surface of the slide several times to remove small glass shards.

Generate the parking slide shown in the top right column of Figure 1 using black ink. Place a large number “9” on a clean slide. Flip the slide over so that the “9” appears as a “P”. Particles can be placed in the loop of the “P”. You may also generate a parking space using a WC scribe within the loop of the “P” if you desire.

General Parking Slide and Axis Slide Models

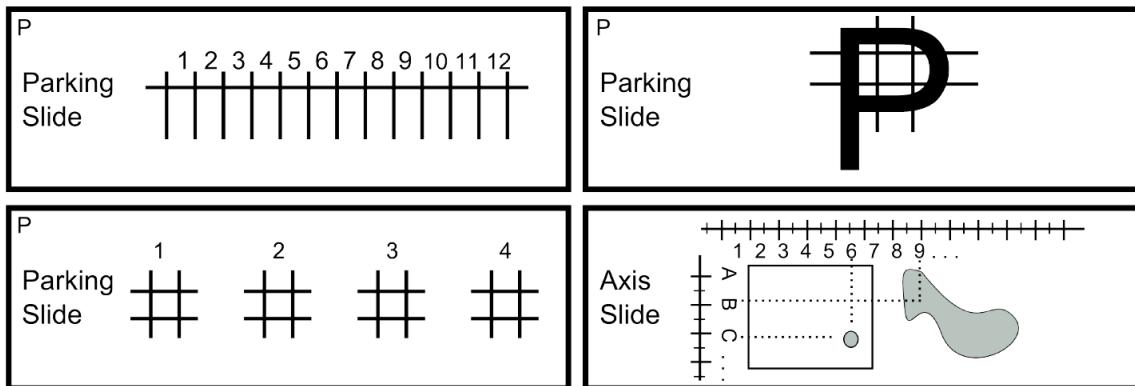


Fig. 1. Parking and Axis Slides.

Once you have generated a parking slide, practice sample handling and micro-manipulation following the instructions below:

1. Sprinkle a few particles of a granular material provided by the instructor (*e.g.*, ground calcite, sand, *etc.*) onto a glass slide.

2. Try to transfer particles to your parking slide.
 - (a) You can increase the tackiness of the needle by “huffing” on it.
 - (b) Touch the needle to the particle, only near the tip of the needle. If the particle travels up the needle toward the handle, it will be difficult to remove the particle from the needle when transferring.
 - (c) To release stubborn particles from the needle’s tip or particles that have traveled up the needle’s length, you will find it helpful to roll the handle of the W-needle holder as you gently press it against the substrate to which you intend to transfer the particle to.
 - (d) Hold the needle at an angle of < 15° relative to the substrate. To obtain this low angle it may be necessary for you to hold the needle by extending your fingers down the length of the handle. At this angle the needle should feel parallel to the working surface. Never stab at the particle you are trying to lift.
 - (e) Be sure to clean your needles often. This is best done by placing a piece of double sided sticky tape on a glass slide. Next, place a drop of water onto the adhesive and then place your needle into the drop of water and against the adhesive on the tape. This will serve to clean the needle. Be sure to clean your needles by observing them with a stereomicroscope.
3. Move the particles as if you are sorting them by “parking” them on your slide.
4. Document this activity and where you parked the particle.
5. Practice this with several particles to become familiar with the technique.

Part II: Physical Characterization of Tapes

The purpose of this exercise is to familiarize the student with taking physical observations and measurements of tape specimens. Record your observations including descriptions and sketches. Recall the procedure for chain of custody and receiving an unknown sample in the notebook guidelines when you are ready to receive the questioned tape sample. Follow the procedure below:

1. Inspect the tape ends of known and question samples for any potential end match. If fracture match is found:
 - (a) Inspect under the microscope and document observations in your notebook.
 - (b) Document the fracture match using photography.
2. Take note of the backing color and any distinctive characteristics in the backing. Is the backing smooth or does it have markings/dimples? If dimples are present describe and sketch the morphology.

3. Look at the color of the adhesive and make a note of it. Is it gray? If so, is it light, medium, or dark gray? If it is white, is it truly white, or is it off-white, or buff (off-white with a slight pink tint)?
4. Measure the width in inches ($\pm \frac{1}{64}$ th of an inch)
5. At one end, gently remove a small portion of adhesive with alcohol and a cotton swab to expose the scrim. Make a note of the textures of the warp yarns and the fill yarns (twisted, filament, or textured). Note the weave pattern. Document what you see with a drawing in your notes.

Create a summary table of your observations. See Table 1 for an example. When complete, return the tape samples to the instructor and affix the chain of custody form to your notebook.

Table 1. Example summary table of physical and chemical characteristics for known and questioned tape samples.

Item ID	T3C21	T3C24-K1	B-V2
Tape Width (n=10)*	48.1 \pm 0.5 mm	48.0 \pm 0.4 mm	48.7 \pm 0.7mm
Backing color	Silver	Silver	Silver
Backing surface	Dimples Spaced 50 \pm 5mm (n=10)	Dimples Spaced 50 \pm 5mm (n=10)	Dimples Spaced 15 \pm 7mm (n=10)
Adhesive color	Cream-clear	Cream-clear	Off-White
Scrim type	Plain weave	Plain weave	Plain weave
Scrim count (warp, n=10)*	20 \pm 0.5 mm	20 \pm 0.5mm	20 \pm 0.5mm
Scrim count (weft, n=10)*	6 \pm 0.3	6 \pm 0.3	8 \pm 0.5
Yarn description	Warp: straight filament Weft: Textured	Warp: straight filament Weft: Textured	Warp: straight filament Weft: Textured

* Note: data represents average measurements and standard deviation from 10 different analysts

Part III: Cross Sectioning of Fibers

High density polyethylene can be used to efficiently generate a quality fiber cross section for materials with high melting points. Although you may elect to cross section any fiber of your choice, you are encouraged to work with fibers that you believe will possess interesting cross sections (such as bi-components, tri-lobial fibers, *etc.*). Keep in mind that this technique requires heat, which can destroy thermo-sensitive fibers. Follow the method outlined below:

1. Trim the end off of a small-bore plastic transfer pipette.
2. Insert the fiber sample into the trimmed fragment.
3. Make a “sandwich” starting with a microscope slide, the trimmed plastic transfer pipette fragment with sample, and finishing with another microscope slide.

4. Heat the “sandwich” on a hot plate, applying pressure to the top microscope slide (using a pencil or other sturdy instrument). Continue heating until the plastic turns clear, confirming that the bore of the pipette has fused together.
5. Remove the slide “sandwich” from the hotplate (be careful...it will be hot!) and let cool. Once cooled, pull the top slide off and transfer the fused pipette tip fragment (with sample) to a plastic slide.
6. Using a Teflon coated razor blade (or other sharp instrument), a stereomicroscope, and a needle, generate several cross sections. Using the needle, appropriately orient and distribute the sections on the slide (and/or transfer each to a glass slide). Examine various sections using a polarizing light microscope. Secure and flatten high quality sections using a glass cover slip.

Submission Requirement for the Lab

Ensure that all activities have been documented in the lab notebook contiguously. Please pay attention to the notebook documentation guidelines; your notebook will be inspected and graded. Additionally, you will need to submit:

1. A one-half page reflection of how comfortable you are with handling of microscopic samples. Be sure to include successes or challenges you experienced. (Part I)
2. Summary table of tape observations. (Part II)
3. Conclusions regarding the tape comparison. (Part II)
4. An image or sketch of your fiber cross section. (Part III)

Lab 2: Vibrational Spectroscopy

Objective of Lab Exercise

The purpose of this lab is to identify unknown samples through the application of vibrational spectroscopy. Examination will be completed on both tape and powder samples utilizing Fourier Transform Infra-Red Spectroscopy. Before proceeding, ensure that the instrument is calibrated by running the proper controls.

Lab Instructions

You will be working as a collective class for this lab exercise. Begin by obtaining known and questioned tape and drug powder samples.

Part I: Analysis of Adhesive Tapes

Begin by examining the questioned samples. Acquire spectra of the backing and adhesive for comparison purposes using the procedures below. Once complete, examine the known samples.

ATR Analysis of Duct Tape Adhesive

1. Clean the crystal gently with a Kimwipe or with a cotton swab dampened with acetone.
2. Collect a background spectrum of air.
3. Position a duct tape sample with the adhesive side against the crystal. The pressure device is not necessary for analysis of the adhesive since it adheres well and provides good contact with the crystal.
4. Collect the adhesive spectrum.
5. Select the peaks (cm^{-1}) and save the spectrum.
6. Search the spectrum against the materials provided in the FTIR lecture to attempt to identify the primary components of the adhesive.

ATR Analysis of Duct Tape Backing

1. Remove the above adhesive sample and clean the crystal gently with a Kimwipe or with a cotton swab dampened with acetone.
2. Collect a blank (background spectrum of air).
3. Gently swab the backing of the duct tape with hexane or methanol to remove any dirt or fingerprints from the area to be analyzed. Position the clean sample with the backing side against the crystal and use the pressure device to apply sufficient light pressure to insure good contact between the sample and crystal.

4. Collect the backing spectrum.
5. Select the peaks (cm^{-1}) and save the spectrum.
6. Search the spectrum against the materials provided in the FTIR lecture to attempt to identify the primary components of the adhesive.
7. Using the above steps, analyze the adhesive side of each the backings (following removal of adhesive and scrim) and compare the results.

Part II: Analysis of Powder Samples

For this portion of the lab, you will examine powder samples using a similar procedure as above. These samples do not need any to undergo any sample preparation, a primary benefit of ATR–FTIR. Begin by examining the questioned samples. Ensure that you clean the instrument thoroughly and run a blank in between each sample to prevent carry over. Once complete, examine the known samples. Save all spectra.

Submission Requirement for the Lab

Ensure that all activities have been documented in the lab notebook contiguously. Please pay attention to the notebook documentation guidelines; your notebook will be inspected and graded. Additionally, you will need to submit two forensic reports¹: one for the examination you conducted on adhesive tapes (Part I), and another for the examination of drug samples (Part II).

¹See the example forensic report on Page 12.

Lab 3: Interpretation of Mass Spectra

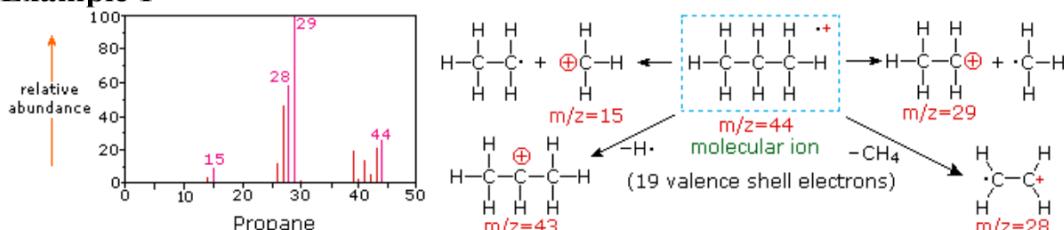
Objective of Lab Exercise

Become familiar with the interpretation of fragmentation patterns in mass spectra (MS). Furthermore, you will practice interpreting MS data through this exercise.

Lab Instructions

The diagram shown in Figure 2 displays the mass spectra of two simple gaseous compounds; propane and cyclopropane. The molecules of these compounds are similar in size, C₃H₈ has a nominal mass of 44 amu, and C₃H₆ has a mass of 42 amu. The molecular ion is the strongest ion in the spectra C₃H₆, and it is moderately strong in propane. The unit mass resolution is readily apparent in these spectra (note the separation of ions having m/z=39, 40, 41 and 42 in the cyclopropane spectrum). Even though these compounds are very similar in size, it is a simple matter to identify them from their individual mass spectra, evident from the partial fragmentation analysis and peak assignment shown in Figure 2. Even with simple compounds like these, it should be noted that it is rarely possible to explain the origin of all the fragment ions in a spectrum.

Example 1



Example 2

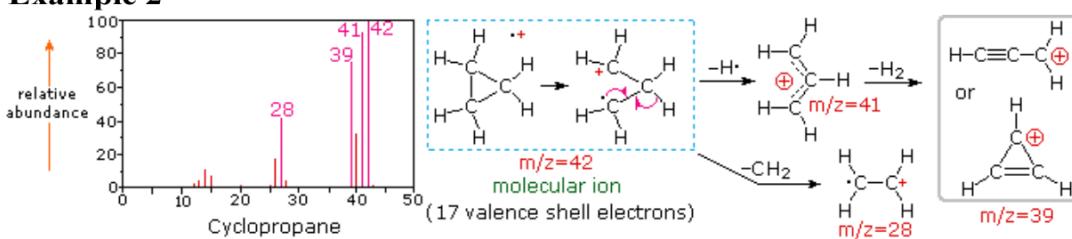


Fig. 2. Parking and Axis Slides.

Part I: Identification of Compounds using MS

Below you will find total ion chromatograms (TICs) and electron ionization MS (gas chromatography/mass spectrometry data) for several compounds. Use the TICs and MS from the known samples to identify all compounds in the questioned samples. Essentially, you are performing a manual library search.

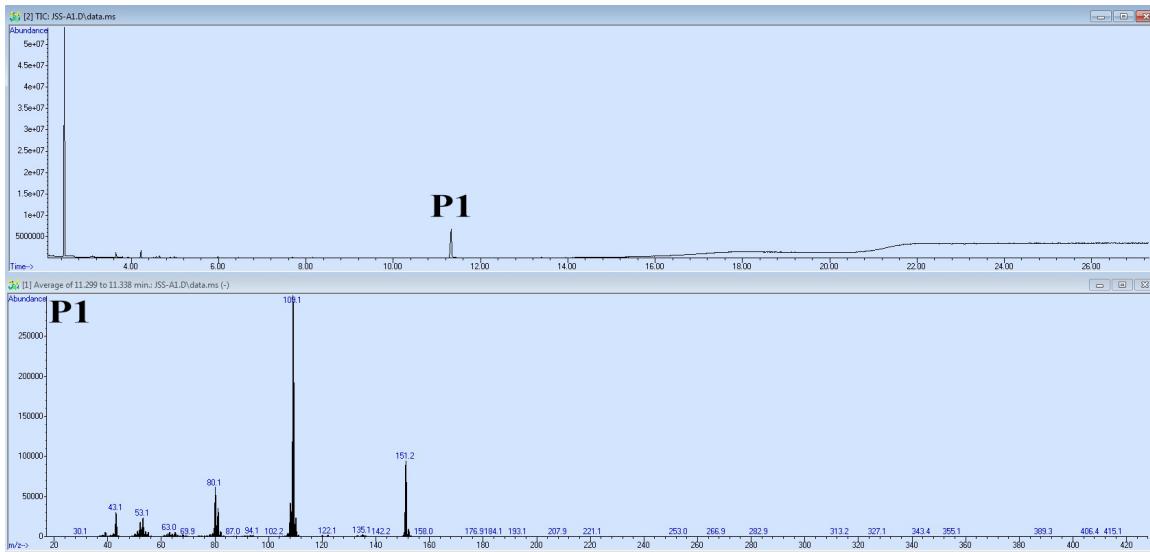


Fig. 3. Questioned Sample 1. Shown is the TIC (top) and the MS (bottom).

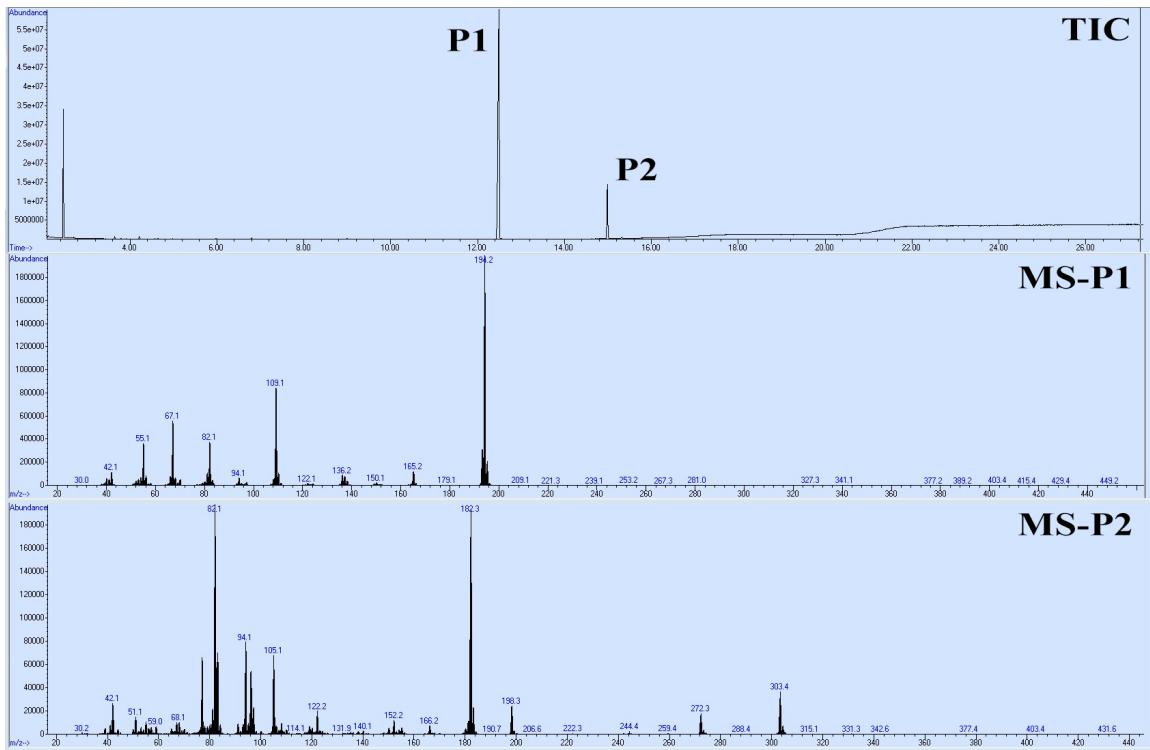


Fig. 4. Questioned Sample 2. Shown is the TIC (top), the MS of the peak at ≈ 11.38 minutes (middle), and the MS of the peak at ≈ 12.49 minutes (bottom).

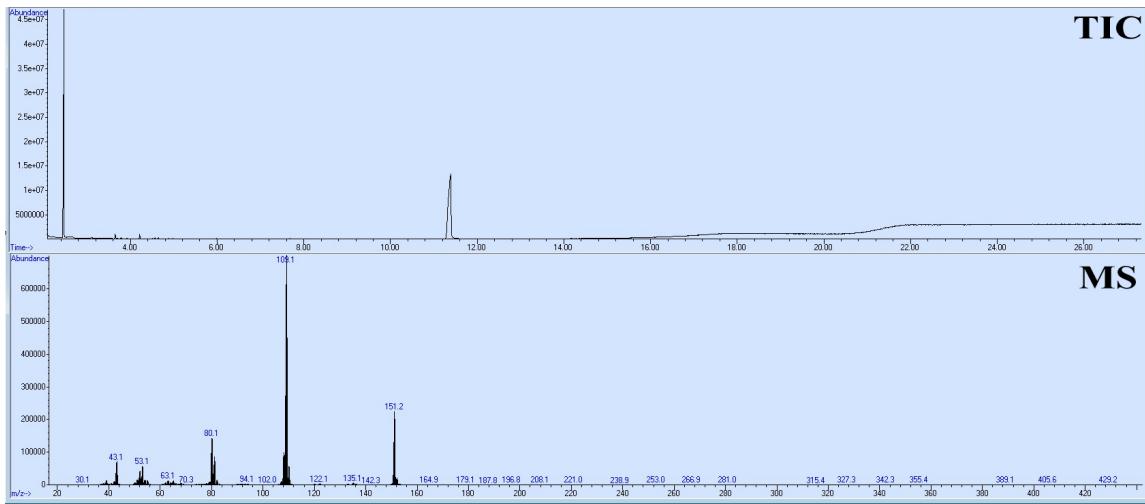


Fig. 5. Known acetaminophen sample. Shown is the TIC (top) and the MS (bottom).

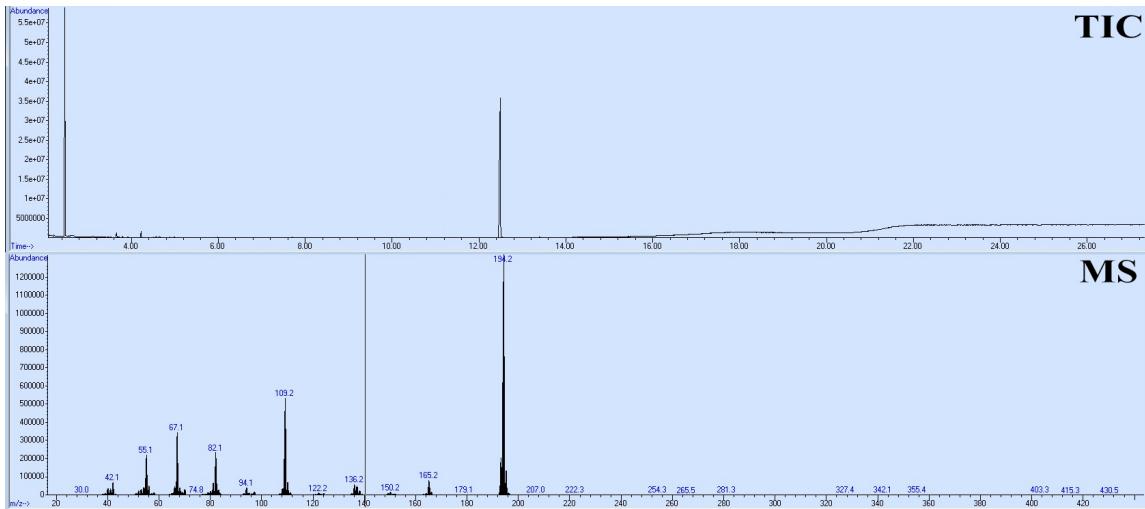


Fig. 6. Known caffeine sample. Shown is the TIC (top) and the MS (bottom).

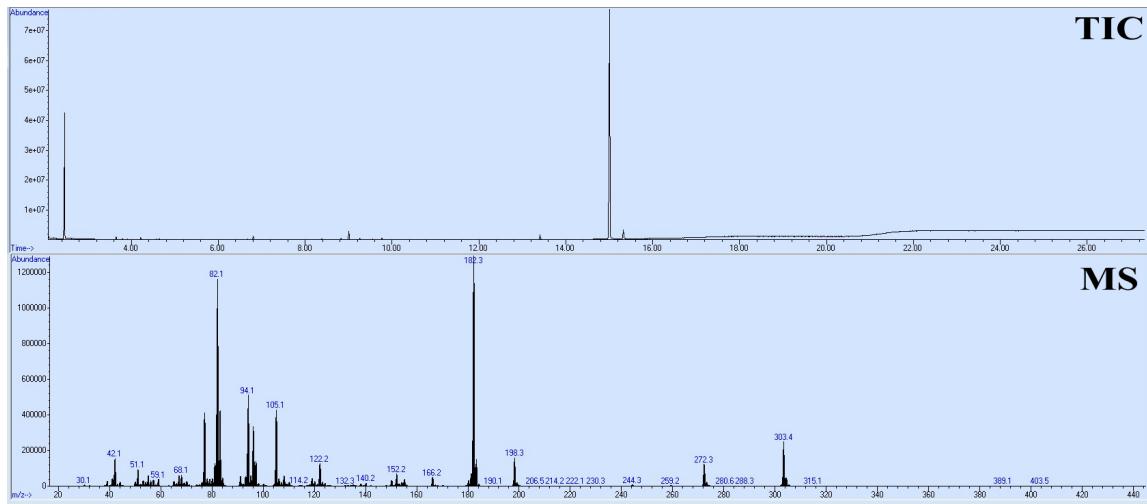


Fig. 7. Known cocaine sample. Shown is the TIC (top) and the MS (bottom).

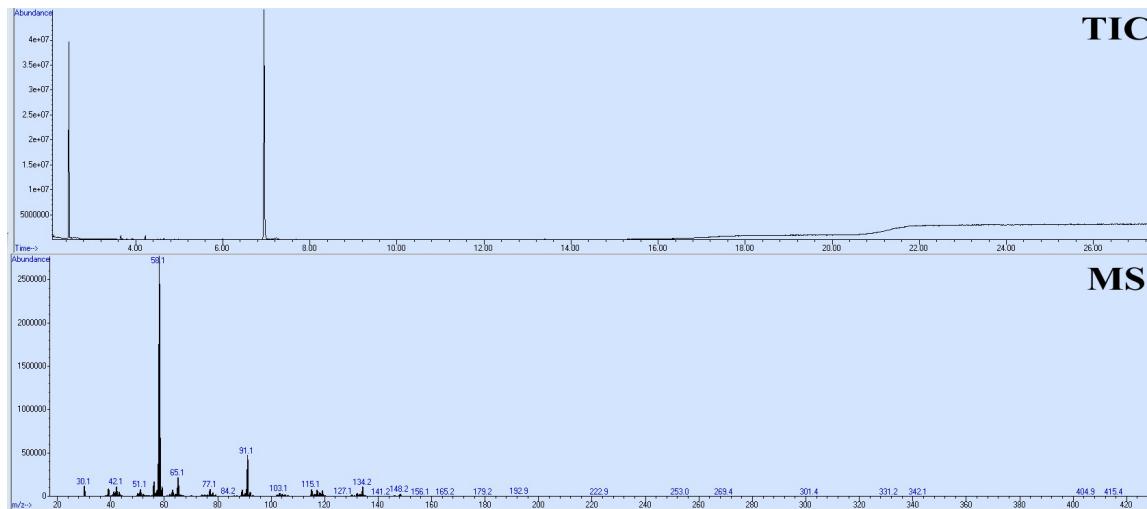


Fig. 8. Known methamphetamine sample. Shown is the TIC (top) and the MS (bottom).

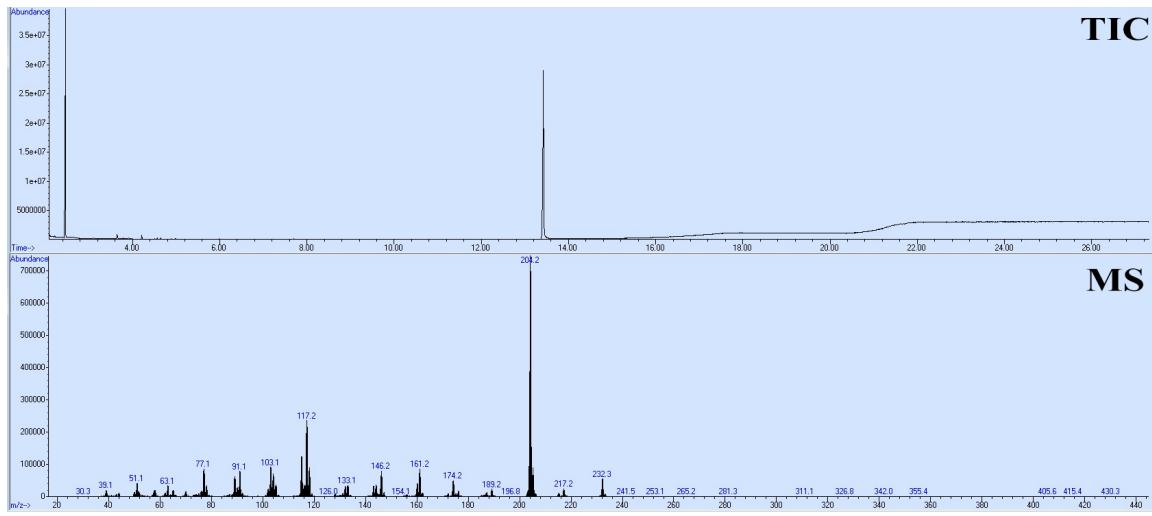


Fig. 9. Known phenobarbital sample. Shown is the TIC (top) and the MS (bottom).

Part II: Examination of Fragmentation Patterns

In this section of the lab, you will explain the fragmentation patterns which create the characteristic MS of cocaine. An example of how this process can be conducted is given below. There are three approaches to analyzing MS peaks in drug samples:

1. Sigma Bond Cleavage
2. Isotopic Contribution Analysis
3. Detailed Fragmentation Analysis – Consideration of Ionization Site

Sigma bond cleavage is most commonly observed in molecules, which can produce stable cations such as saturated alkanes, secondary and tertiary carbocations. This occurs when an alpha electron is removed. The C–C bond elongates and weakens causing fragmentation. Fragmentation at this site produces a charged and a neutral fragment. An example of a sigma bond cleavage reaction (σ -cleavage) is shown in Figure 10.

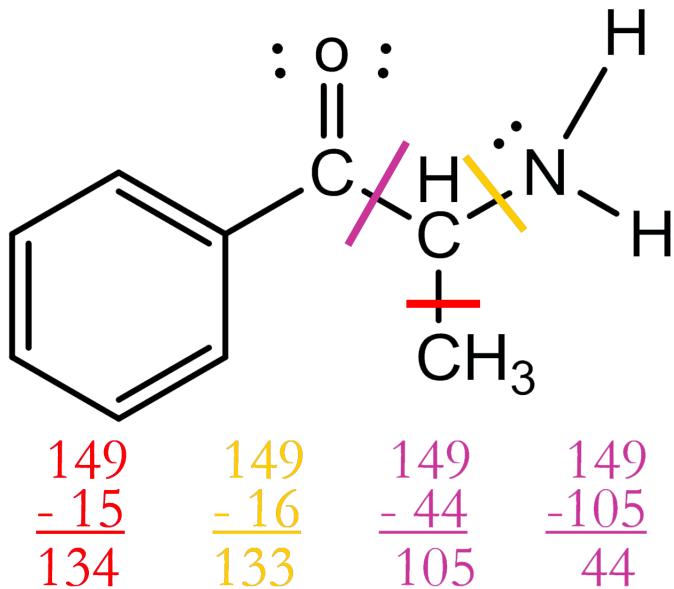


Fig. 10. Example σ -cleavage and the resultant fragment masses.

The second approach is isotopic contribution analysis. More information is available from: McLafferty and Turecek “Interpretation of Mass Spectra” 4th edition (1993), Ch. 2. Recall that the ease of ionization is as follows: non-bonding electrons > π electrons > σ bonds.

- Determine elemental composition of fragments based on isotope peak ratios
- Pick peak of interest ‘X’
- Assign elements with X+2 isotopic contribution (Except O)
- Assign elements with X+1 isotopic contribution
- Assign number of O
- Determine number of rings and double bonds
- Determine empirical formula, then propose structure(s)
- Does the structure make sense?

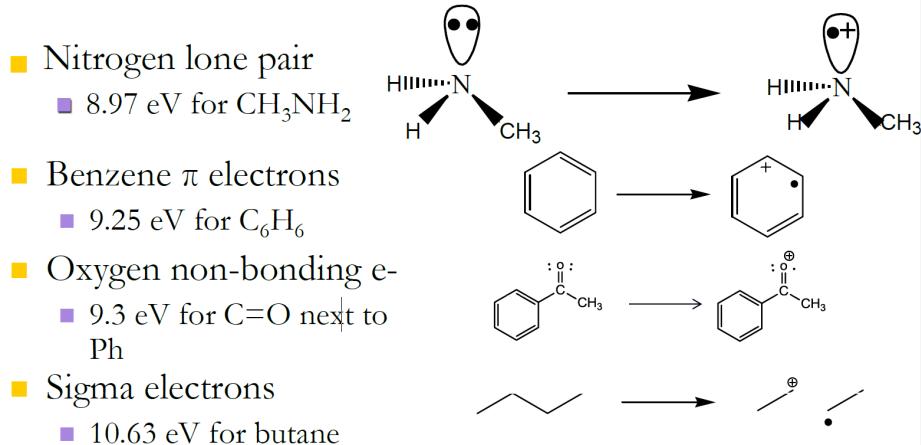


Fig. 11. Ionization potentials.

Also note that homolytic cleavage occurs with radical cations.

- Occurs with radical cations
- Initiation occurs at the radical site.
- The charge is retained on the original site
- Movement of single electrons characteristic
- $\text{OE} \rightarrow \text{radical} + \text{EE}$
- Tendency for homolytic cleavage: $\text{N} > \text{O}, \text{S}, \pi, \text{R}\cdot > \text{Cl}, \text{Br} > \text{H}$

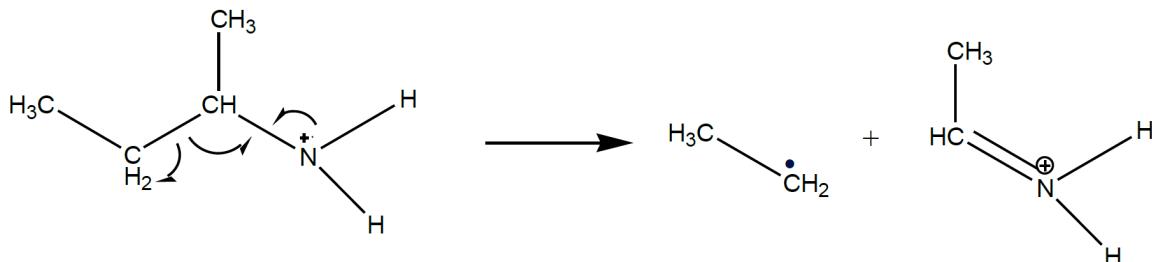


Fig. 12. Diagram illustrating homolytic cleavage.

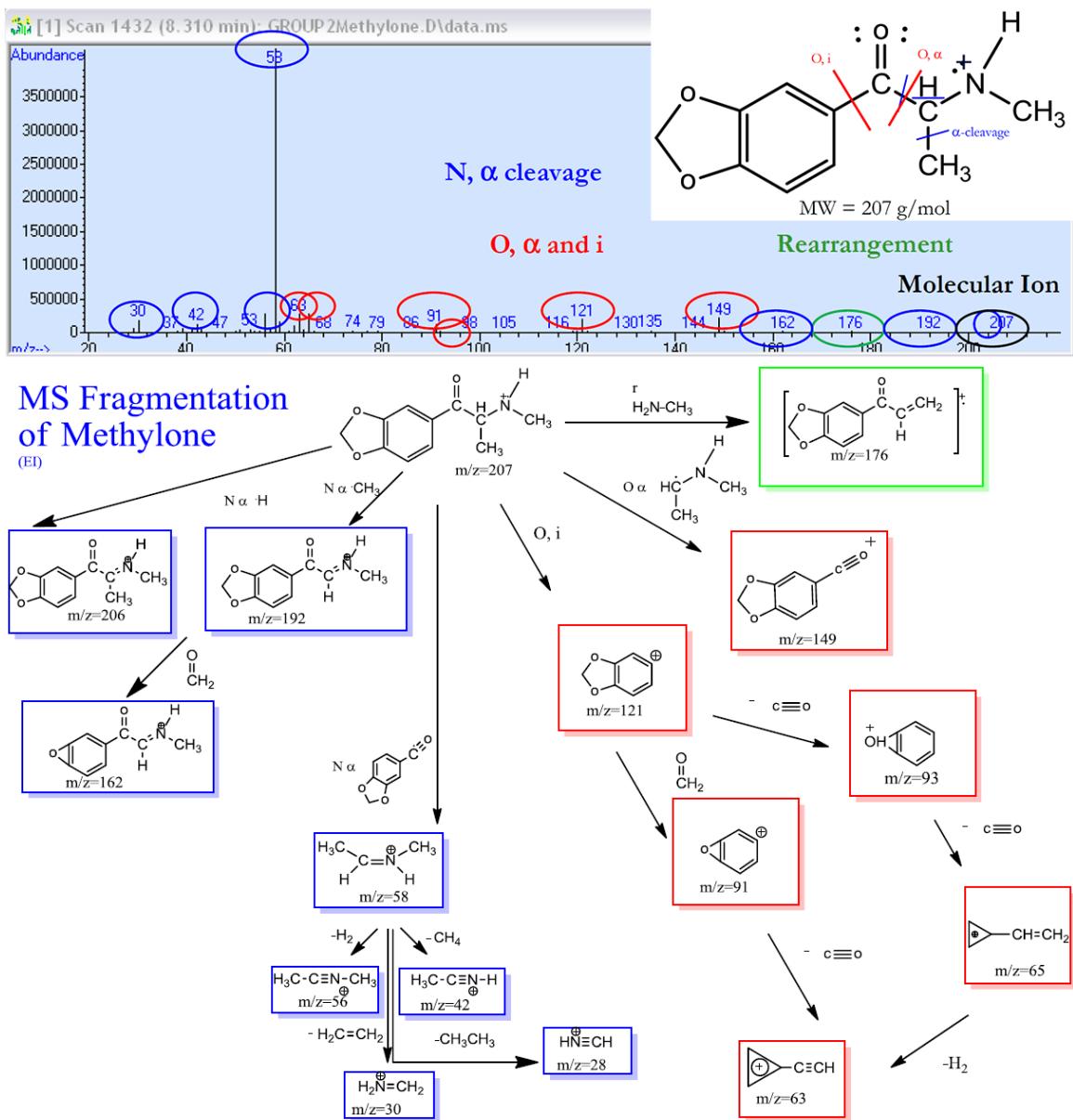


Fig. 13. Fragmentation of the “bath salt” designer drug 3, 4-methylenedioxymethcathinone, commonly known as methylene.

Following the example given in Figure 13, complete this process for cocaine. The MS for cocaine is given in Figure 14².

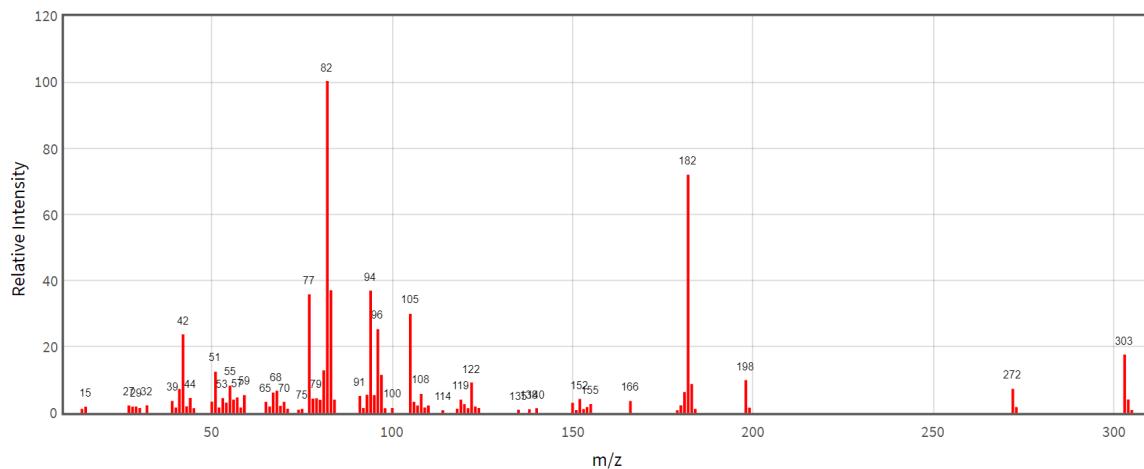


Fig. 14. MS of cocaine. Acquired from the NIST Mass Spectrometry Data Center.

Submission Requirement for the Lab

There is no lab notebook requirement for this lab. You will need to submit a forensic report for Part I of the lab. Author the report as if you had conducted the analyses using GC/MS and state your conclusions. The parameters used for the Agilent 7890B gas chromatograph and the Agilent 5977A mass spectrometer detector during this analysis are given in Table 2. Refer to these instruments and parameters in your report.

Table 2. GC/MS parameters used when acquiring data for this lab.

GC Parameters		MS Parameters	
Inlet Temp	250°C	Transfer Line	280°C
Injection Volume	1ul	Solvent Delay	2 mins
Split Ratio	Splitless	Ionization Energy	70 eV
Carrier Gas	He	Mass Energy	30-450
Column	DB-5MS 30m x 0.25mm x 0.25mm		
Initial Temp	50°C hold 2 mins		
Ramp	15°C /min		
Final Temp	280°C hold 3 mins		

Additionally, you will need to submit your completed fragmentation pathway diagram for cocaine from Part II.

²The following article may be of great help: Smith, RM. and Casale, JF. "The Mass Spectrum of Cocaine: Deuterium Labeling and MS/MS Studies." *Microgram Journal*, **2010**.

Lab 4: Color Presumptive Tests

Objective of Lab Exercise

Become familiar with how presumptive color tests can be performed in the laboratory to direct collection and subsequent analysis of evidence.

Lab Instructions

Reagents will be prepared for you. The reagents and tests to be performed are given in the paper by O'Neal *et al.* provided on Canvas (C.L. O'Neal, D.J. Crouch, A.A. Fatah, Validation of twelve chemical spot tests for the detection of drugs of abuse, *Forensic Science International*, 109 (2000) 189-201). When completing presumptive color tests in sequence for the identification of a substance, refer to Figure 16 from R.H. Liu, D.E. Gadzala, Handbook of Drug Analysis, ACS, Washington, DC, 1997.

All reagents and samples for the color tests are provided in Table 3.

Table 3. Reagents and samples for the lab exercise.

Reagents	Samples
	Acetaminophen
	Aspirin
	Excedrin
Marquis Reagent	Heroin (1mg/mL Solution)
Mandelin Reagent	Lidocaine
Nitric Acid	Procaine
Scott's Reagent – $Co(SCN)_2$	Sugar
	<i>Unknown #1</i>
	<i>Unknown #2</i>
	<i>Unknown #3</i>
	<i>Unknown #4</i>

Label all wells before placing any samples in them and draw the well plate in your notebook and label each well there as well. Be sure you can define the orientation of the well plate if it is symmetrical to ensure that the results you obtain are accurate. Orientation can be maintained by leaving at least one corner well empty (if needed). Next, place a few crystals of each sample in a clean well in a spot-plate. Add one or two drops of reagent as specified in each test. Observe your results and photograph, if possible. See Figure 15 for an example well plate being used for color tests (note that the expected reaction colors are approximate). Compare your observations with the expected results outlined in the article and draw conclusions for each sample. Document completely in your notebook.

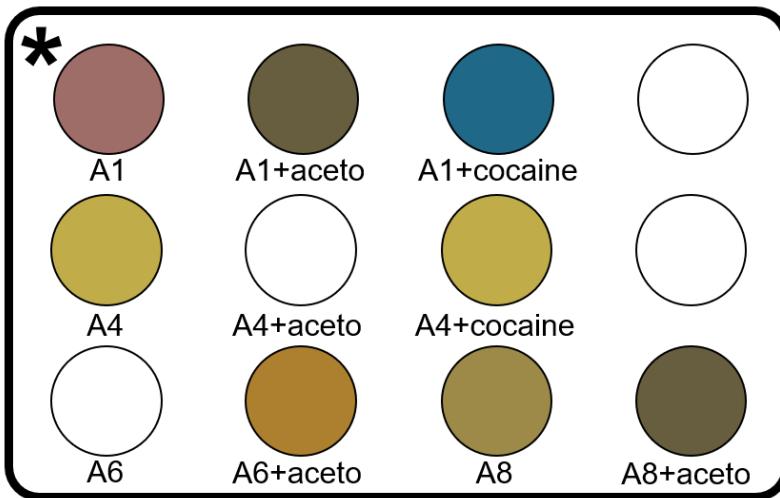


Fig. 15. Example layout of a well plate for color tests and drugs. Note the * symbol in the upper left corner for orientation purposes and that each well in use is labeled.

Submission Requirement for the Lab

Ensure that all activities have been documented in the lab notebook contiguously. Please pay attention to the notebook documentation guidelines; your notebook will be inspected and graded. Additionally, you will need to submit a forensic report for your analysis.

Table 3.2. Flowchart for multiple and sequential use of reagents for color tests.^a

First reagent ^b		Second reagent		Third reagent		Fourth reagent	
Color	Color	Drug ^c	Color	Drug	Color	Drug	Color
Marquis	No color	p-DMAFB	Green Olive-brown	Mandelin	No color	HNO ₃	Amphetamines Mescaline
Orange-brown	No color		No color		No color	Co(SCN) ₂	Sugar Demerol
Purple	Purple	LSD	Co(SCN) ₂				
Yellow-green	No color Greenish-blue/blue	STP Marezine, Ritalin					
Pink-red	No color Greenish-blue/blue	Aspirin, Contac, Dristan, Excedrin Methadone, phencyclidine	Co(SCN) ₂				
No color	No color	Co(SCN) ₂					
Purple-violet-black	Purple Olive Pale blue	Methapyrilene•HCl Codeine, opium MDA	HNO ₃				
Tan	Purple	Dille-Koppanyi					

^a Abbreviations: n-DMAR: *n*-dimethylaminobenzaldehyde; I: SD: l-isoserine acid diethylamide; MDA: 3,4-methylenedioxymethamphetamine; STP: 2-(*S*)-dimethylamino-4-methylamphetamine.

See Table 3.1 for reagent formulas.

^c Abbreviations: *p*-DMAB; *p*-dimethylaminobiphenyl.

^c Abbreviations: *n*-DMAB: *n*-dimethylaminobenzaldehyde; I SD: *l*-seric acid.

^c Abbreviations: *p*-DMAB; *p*-dimethylaminobenzaldehyde; LSD: lysergic acid

Fig. 16. Flowchart of multiple and sequential use of reagents for color tests.

Lab 5: Micro-chemical Tests

Objective of Lab Exercise

In this exercise students will learn the utility of microchemical crystal tests. These techniques require very little sample, and can be utilized to detect various materials, including inorganic ions, metals and drugs of abuse. Often, rapid and simple tests are used first to determine the likely drug present before more detailed instrumental analysis is conducted to confirm the identity of the drug. When used as part of an analytical scheme for drug identification, microcrystalline tests are considered to be selective tests; that is, they are used to indicate the likely drug present, but these tests alone cannot definitively identify the drug.

Lab Instructions

In general, microchemical crystal tests can be regarded as the application of known reagents to small amounts of unknown material to form microcrystalline derivatives with well characterized morphologies and/or optical properties. The reactions are carried out on a drop-scale on microscope slides and are usually studied with a low power (100X) objective-ocular combination. The most authoritative reference text for the detection of inorganic ions is the **Handbook of Chemical Microscopy, Vol II**, (1989) by Chamot and Mason. This text describes general techniques, specific reactions and descriptions of resulting microcrystals (including numerous photomicrographs).

Within this field, the term test drop refers to a drop of solution containing the substance being tested. This drop (or a product of this drop, such as a vapor), is brought into contact with what is referred to as the **reagent drop**, which is a solution containing a known material with the potential to react with the test drop. In general, Chamot and Mason (1989) suggest that the test drop should be thinner and taller than the smaller and higher reagent drop.

The first chapter in the *Handbook of Chemical Microscopy* by Chamot and Mason (1989) describes ten different possible methods that can be used to combine test and reagent drops, specifically designed to facilitate microcrystal formation. In Method I (Figure 17), the more concentrated reagent drop is caused to flow and sink into the test drop. To accomplish this, a microspatula (toothpick) is drawn from the reagent drop to the test drop, forming a narrow channel between the two drops. The channel is created using a single stroke, without mixing, generating a concentration gradient between the two drops. Ideally, there will be a concentration within this gradient that is optimal for crystal formation. Method II (Figure 17) is also very useful, which involves the addition of a fragment of solid reagent to the test drop, ensuring an excess of reagent during the initial stages of the reaction. Again, the reaction is not stirred, but allowed to progress as the concentration gradient changes in the center of the test drop toward the periphery.

Another useful procedure is referred to as **Method IX**, which involves the evolution, capture and reaction between a gas and reagent. The test drop (and any required liberating

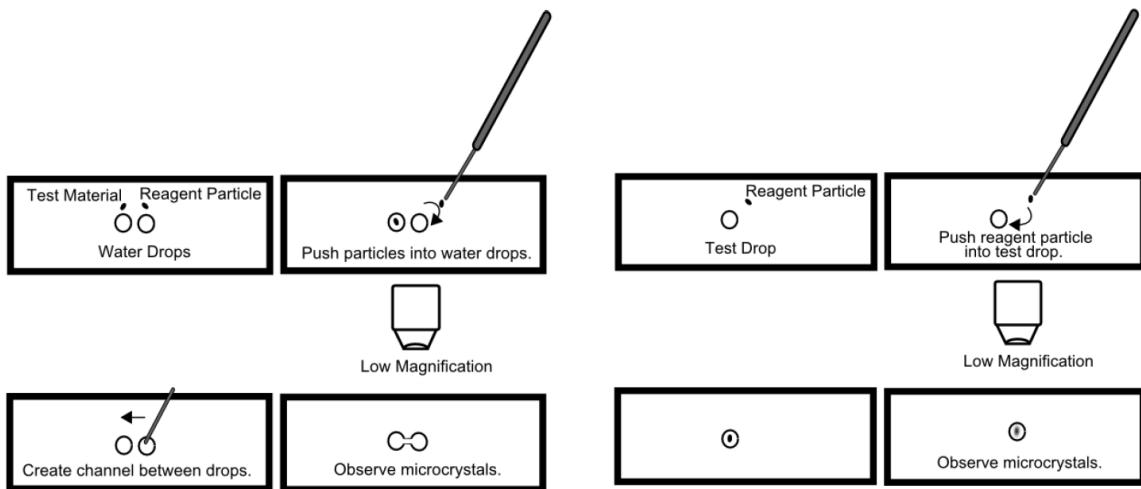


Fig. 17. The leftmost image depicts Method I and the rightmost image depicts Method II.

materials) are added to a small crucible that is covered with a microscope slide containing what is referred to as a hanging drop (or a trapping/fixing reagent that adheres to the underside of the slide, closest to the evolving gas). After the slide with the hanging drop is in place, the sample is heated gently (avoid spattering!) to help evolve gaseous products that will be trapped by the hanging drop.

Flat wooden toothpicks serve nicely as disposable microspatulas, but the analyst must keep in mind that microchemical reactions are extremely sensitive, and as such, precautions must be taken to guard against contamination. All equipment (*e.g.*, slides, spot plate, and microspatulas, *etc.*) should be scrupulously cleaned PRIOR AND AFTER use. Microscope slides (even those labeled as “precleaned”) should be cleaned prior to use, and it is never advisable to reuse micropipet tips. In addition, make sure your workspace is clean and organized; be sure to label slides as necessary to avoid confusion.

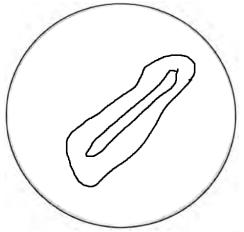
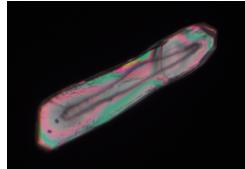
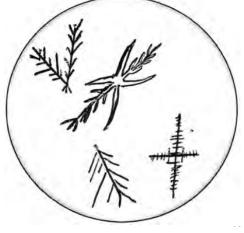
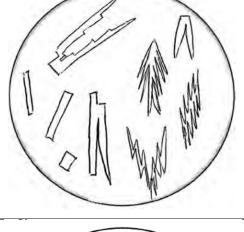
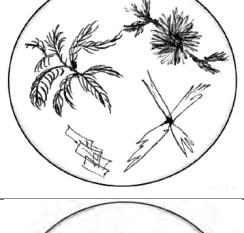
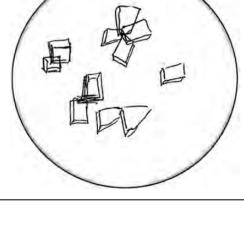
Exercise

You will be performing microcrystal tests on the following samples: acetaminophen, cocaine, methamphetamine, phosphates, and sulfates.

1. In groups of two, obtain and clean a spot plate.
2. Each group will also require a paper towel, a bottle of distilled water, microscope slides, and transfer pipettes.
3. Carefully label the spot plate, giving a single well to each sample and reagent. You will need very little material for this reaction, so begin with extremely dilute samples (it is straight-forward to increase the concentration by adding more solid, or to decrease the concentration by adding more distilled water).
4. Label the transfer pipettes using the same system you use to label the spot plate, and lay each on the paper towel but be sure they do not touch.

5. Conduct tests one by one, alternating lab partners, as outlined in Table 4
6. Each test must be appropriately described and sketched in the notebook.

Table 4. Summary table of microcrystal tests.

Sample Tested	Method	Reagent (Drop)	Morphology	Crystals
Acetaminophen	II	AuCl Solution		
Cocaine	I	AuCl Solution		
Methamphetamine	I	PtCl Solution		
Phosphate – PO_4^{3-}	II	Silver Nitrate Solution		
Sulfate – SO_4^{2-}	II	Silver Nitrate Solution		

After you have mastered all techniques, prepare a summary table of all the tests conducted. This table will include a column for the sample, the reagents used, the methods used, the result/reaction, and the page number(s) in your notebook where the original observations are located. Clearly label the table as a summary and use appropriate underlined

column headings. Alternatively, cut out and tape the following table into your lab book, and annotate it appropriately to include the page numbers in your notebook where each test can be found. Whatever you decide, this must happen before the formal completion/report page, and before leaving the laboratory.

Submission Requirement for the Lab

Ensure that all activities have been documented in the lab notebook contiguously. Please pay attention to the notebook documentation guidelines; your notebook will be inspected and graded. Additionally, you will need to submit:

1. Completed summary table
2. A one-half page reflection of this exercise

Lab 6: Scanning Electron Microscopy

Objective of Lab Exercise

Learn the fundamentals scanning electron microscopy (SEM) and how it can be utilized for elemental analysis of forensic evidence.

Lab Instructions

This lab will consist of a demonstration of SEM. It is imperative that you review the lecture materials so that you are familiar with how the SEM functions. Questions regarding detectors, backscattered and secondary electrons may be asked throughout the demonstration. SEM can be used to image the sample and energy dispersive X-ray spectroscopy (EDS) can accompany this technique through elemental analysis. An example image of a paint chip under SEM is given in Figure 18. The spectrum detailing the elemental composition analysis with SEM-EDS of the base coat layer of the same paint fragment is given in Figure 19.

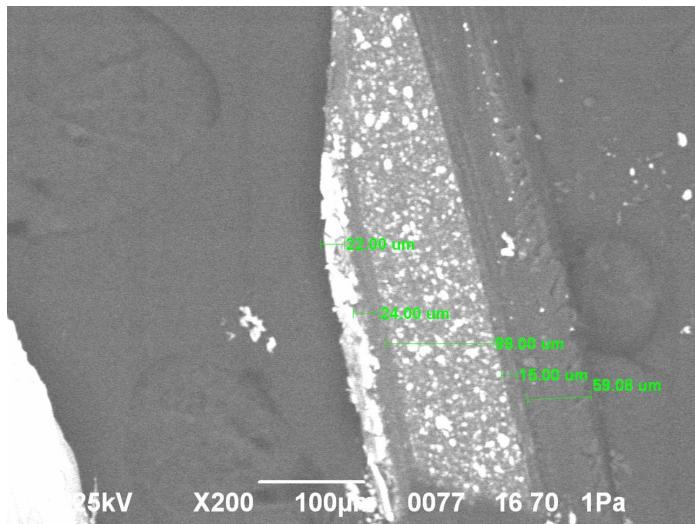


Fig. 18. A cross sectional image of a paint fragment. This image was taken with the JEOL SEM at 200X magnification and a measurement of each layer thickness is provided.

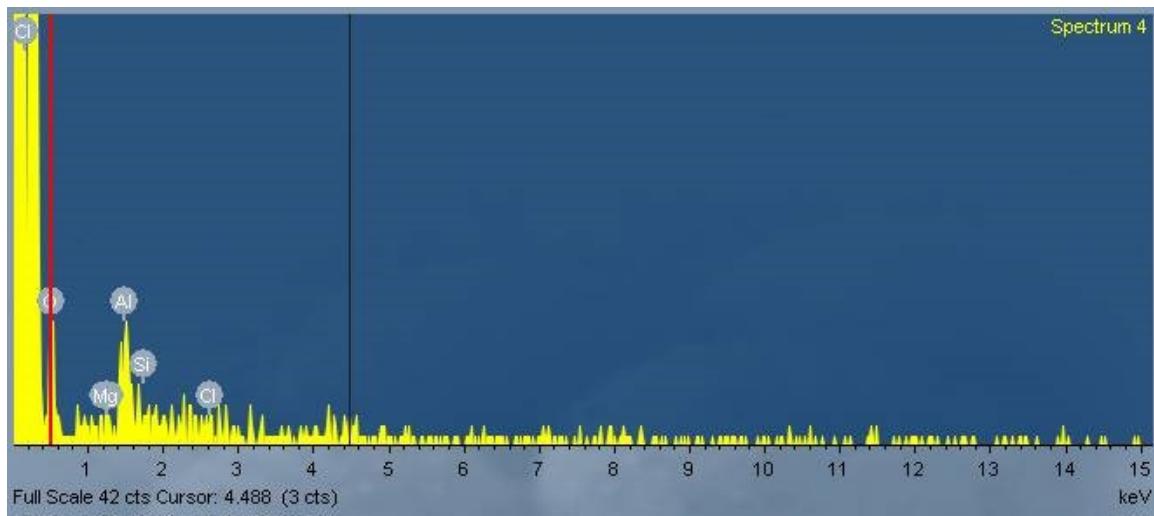


Fig. 19. Elemental Composition Collected from the Base Coat Layer of the paint fragment using SEM-EDS. Major peaks are identified and labelled as shown.

Submission Requirement for the Lab

A one page reflection about the usage of SEM. Be sure to comment about SEM as an imaging technique and as a method for elemental analysis.

Lab 7: Laser Ablation Methods for Trace Analysis

Objective of Lab Exercise

Become familiar with the discrimination abilities of laser ablation inductively coupled plasma mass spectrometry (LA–ICP–MS). Furthermore, you will conduct data analysis on the results to reach a source conclusion about a sample.

Lab Instructions

LA–ICP–MS is one of the most exciting analytical technologies available because it can perform ultra-highly sensitive chemical analysis down to ppb (parts per billion) level – without any sample preparation. Samples can be both conducting or non-conducting, and the analysis can be performed in the air without the need for a complex vacuum system. Results are available within seconds; therefore LA–ICP–MS delivers that fastest analysis speed of all analytical techniques with the limit of detection approaching ppb level.

The sample mass size required for LA–ICP–MS analysis is sub-microscale – picograms to femtograms. Traditional liquid nebulization approaches for ICP–MS require the removal of milligrams of sample mass in order to be effective. The two most commonly used laser-based methods are bulk analysis with a typical laser spot size of $100 \sim 350\mu\text{m}$ and micro-analysis with the laser spot size as small as a few microns. When applied with optimized laser ablation conditions and ICP–MS data acquisition protocols, LA–ICP–MS allows versatile solid sampling schemes that include:

- Bulk analysis
- Local inclusion and defect analysis
- Depth profiling
- Elemental/isotope mapping

To conduct the analysis, a laser ablates a small spot on the surface of the analyte. Ablation is a progressive and superficial destruction of a material by melting, fusion, sublimation, erosion and explosion. The removed or ablated material is then transported to the ICP via nebulizer gas. The inductively coupled plasma atomizes the ablated sample and creates atomic and small polyatomic ions. The ionized sample is then detected using mass spectrometry as discussed earlier this semester. A general schematic of a LA–ICP–MS system is given in Figure 20.

You will be provided with a spreadsheet of data acquired from the analysis of a known glass sample (3 spots, 4 ablations each) and a questioned glass sample (3 fragments, 3 ablations each). This analysis includes the following elements: Mg25, Al27, K39, Ca42, Ti49, Mn55, Fe57, Rb85, Sr88, Zr90, Ba137, La139, Ce140, Nd146, Hf178, Pb208. Images of the glass fragments analyzed are shown in Figure 21.

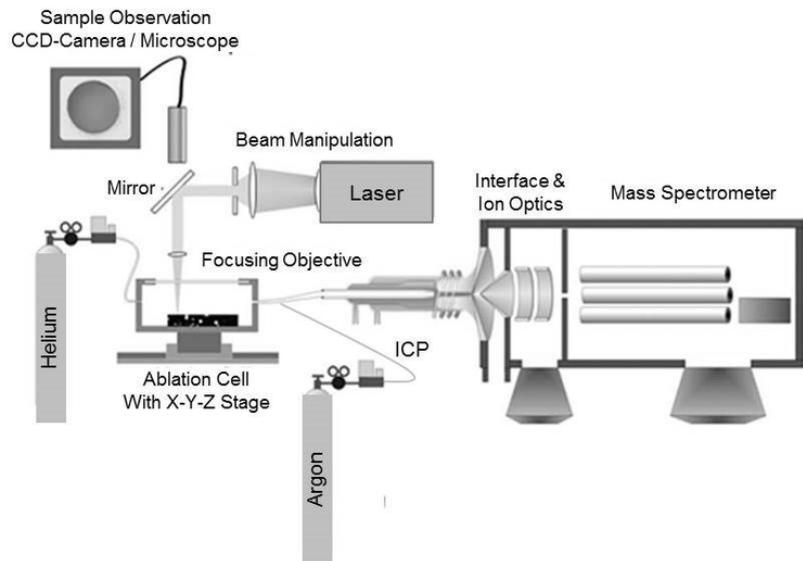


Fig. 20. Elemental Composition Collected from the Base Coat Layer of the paint fragment using SEM-EDS. Major peaks are identified and labelled as shown.

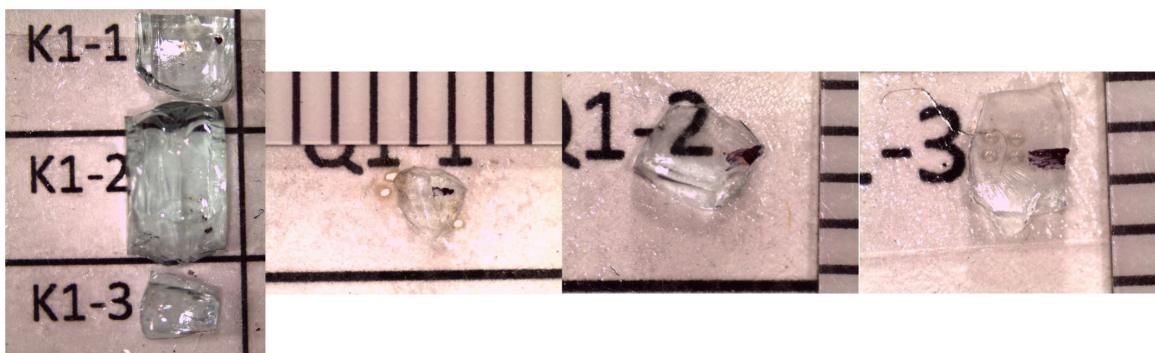


Fig. 21. Shown are the glass fragments (K1-1, K1-2, and K1-3) collected from the victim's residence (left) and the questioned glass fragments (Q1-1, Q1-2, and Q1-3) which were recovered from a suspect's clothing (right).

Begin by calculating the mean, standard deviation, and %RSD for each element of the known sample. Next find the mean of each questioned fragment and determine whether it is within ± 4 standard deviations of the known average. If the questioned sample is within ± 4 standard deviations, it cannot be discriminated from the known sample.

Next, make bar plots which show the mean intensities of each sample. The known sample should have error bars present. These plots will compare the element intensity per sample relative to the known and the error bars show the bounds by which the sample can be distinguished. *Note: it would be best to group elements of similar intensities and create inset plots.*

Submission Requirement for the Lab

There is no lab notebook requirement for this lab. You will need to submit a forensic report for your analysis. Author the report as if you had conducted the analyses using LA-ICP-MS and articulate your conclusions. Include the necessary figures/plots to explain your results.

Lab 8: Casework Practicum

Objective of Lab Exercise

This assignment is meant to serve as a practical application of the information learned throughout this course to a mock criminal investigation. Students will examine the analytical results of items of evidence, draw conclusions, and author a forensic report for a mock case. This casework practicum is designed to closely mimic casework and the examinations that forensic practitioners complete daily.

This exercise aims to promote effective teamwork and peer discussions; therefore, students should discuss and select the strategies for analysis and interpretation of the case. Each team should be prepared to identify additional sources to support their opinions. Main exercises will be conducted in class; however, it is expected the team members meet outside of the regular class schedule.

By the completion of the practice the students should be able to understand and explain:

- The type(s) of evidence recovered and relevance in the case
- Detailed description of the selected analytical scheme
- Detailed physical characteristics and chemical composition of the items
- Data interpretation and conclusions of the case study

Lab Instructions

You will receive a list of evidence collected by the crime scene investigation team with a completed request for analysis submission form. Based on the case study, evidence received, requests for analysis, and the laboratory results you will interpret the findings for the case scenario. Begin with a discussion amongst your team of the analytical strategy for the examination of the evidence submitted for analysis and whether it provides appropriate data for the interpretation of your evidence. The team will then conduct any data analysis and interpretation and have discussions about the report and conclusions derived from the examinations.

The team should prepare a written forensic report (with appendix of technical data, summary tables) which documents the examination, results, and conclusions drawn for your case.

Case Scenario – Incident No. 21-12011309

Stella ARTOIS was the victim of a hit and run incident at the intersection of Snelling and Hewitt Avenues in Saint Paul, MN 55104 at approximately 2000 hours. She was found unresponsive and was later pronounced deceased at the scene by the Ramsey County Medical Examiner at 2058 hours. ARTOIS was reported to have an apparent deep laceration to the left side of her head and her left hand had numerous abrasions. Tire marks and glass fragments were found on the street which are indicative of a hit and run scenario.

The CSI team recovered the following evidence from the scene at the intersection of Snelling and Hewitt Avenues:

- Item Q1 – 10 fragments of glass collected from the road near the ARTOIS's body
- Item Q2a & Item Q2b – Glass fragments collected from the scalp and hair of ARTOIS
- Item Q3a & Item Q3b – Paint chips collected from the scalp and hair of ARTOIS
- Item Q4a (shirt), Item Q4b (shorts), Item Q4c (shoes), Item Q4d (socks) – Clothing from the ARTOIS
- Item Q5 – Hair specimens from the head of ARTOIS
- Item Q6 – Blood sample (swab from open laceration) from the ARTOIS
- Item Q7a & Item Q7b – Photographs and measurements of tire markings



HAMLINE
UNIVERSITY

Hamline University Forensics Science Program
1536 Hewitt Ave
Saint Paul, MN 55104-1284

**Forensic Laboratory
Case Submission Form**

PLEASE TYPE OR PRINT LEGIBLY

Agency Case No.: 21-12011309

Sex Crime Kit Tracking No.: N/A

Evidence No. (HU Det Use): _____

Submitting Agency: Saint Paul Police Department

Date: 12/01/2021

Mailing Address: 367 Grove St.

City: Saint Paul

ZIP: 55101

Investigator: Evan Williams

Title: PFC

Email: bourbon@mail.sppd.org

Phone #1: 651-291-1111

Phone #2: _____

Criminal Offense: Manslaughter/Hit and Run

Incident Date: 12/01/2021

Time: 2000

County of Offense: Ramsey

Brief Description of Crime: Victim found unresponsive after being hit by a vehicle - evident from tire marks and glass fragments proximal to body

List Items Submitted:

1. Item Q1
2. Item Q2a
3. Item Q2b
4. Item Q3a
5. Item Q3b
6. Item Q4
7. Item Q5
8. Item Q6
9. Item Q7
10. _____

List Sections(s) and Examinations Requested:

- | |
|--|
| Forensic Chem/Trace: Glass analysis |
| Forensic Chem/Trace: Glass analysis |
| Forensic Chem/Trace: Glass analysis |
| Forensic Chem/Trace: Paint analysis and PDQ query |
| Forensic Chem/Trace: Paint analysis and PDQ query |
| Evidence Intake: Search for trace evidence |
| Forensic Chem/Trace: Hair examination (retain) |
| Forensic Bio/DNA: Control DNA Sample - Victim (retain) |
| CSI: Crash Reconstruction |

1) Victim: Stella Artois Race: W DOB: 10-10-1995 SSN: 867 - 53 - 0909

2) Victim: _____ Race: _____ DOB: _____ SSN: _____ - _____ - _____

1) Suspect: _____ DOB: _____ SSN: _____ - _____ - _____

SID No: _____ FBI No.: _____ Race: _____ Sex: _____ Height: _____ ft. _____ in. Wt.: _____ lbs.

2) Suspect: _____ DOB: _____ SSN: _____ - _____ - _____

SID No: _____ FBI No.: _____ Race: _____ Sex: _____ Height: _____ ft. _____ in. Wt.: _____ lbs.

FOR LABORATORY PERSONNEL USE ONLY - DO NOT WRITE IN THIS BOX

Received via: Evidence Locker U.S. Mail Certified Mail _____
 Other _____

Date: _____ / _____ /20_____

Laboratory Case No.: _____ Request No.: _____

Two copies: Submit with evidence

One copy: Retained by submitting officer

Completed Analysis by Evidence Type

The following analytical scheme (methods italicized) have been conducted on the **GLASS** samples and the results are provided on the ensuing pages:

1. *Optical microscopy/Physical measurements*: images provided
2. Refractive Index: *Glass Refractive Index Measurement System (GRIM)*
3. Elemental Analysis: *Laser Induced Breakdown Spectroscopy (LIBS)*

The following analytical scheme (methods italicized) have been conducted on the **PAINT** samples and the results are provided on the ensuing pages:

1. *Optical microscopy/Physical measurements*: analyst notes provided
2. Presumptive Testing: *Microchemical Solubility Tests*
3. Elemental Analysis: *Micro-Fourier Transform Infra-Red Spectroscopy (μ -FTIR)*
4. Elemental Analysis: *Scanning Electron Microscopy – Energy Dispersive X-Ray Spectroscopy (SEM-EDS)*
5. Database Inquiry: *Paint Data Query (PDQ) database search*

Paint Analytical Results:

After examination of the PAINT chip samples (Item Q3a & Item Q3b), the following results were returned from the *PDQ database*:

1. Dark green Jeep Compass Latitude 2015
2. Green Jeep Grand Cherokee Limited Edition 2015
3. Green Jeep Cherokee Trail Hawk 2015
4. Green Jeep Compass Latitude 2014
5. Green Jeep Compass Latitude Limited Edition 2014

From this database information, police located two potential suspect vehicles with front end damage at local auto-body repair shops.

- Dark green Jeep Compass Latitude 2015 – Vehicle 1
- Green Jeep Cherokee Trail Hawk 2015 – Vehicle 2

Additional paint evidence was recovered from these vehicles: Item 1 – paint sample from damaged hood of Vehicle 1 (Jeep Compass Latitude 2015) and Item 2 – paint sample from damaged hood of Vehicle 2 (Jeep Cherokee Trail Hawk 2015).

Optical microscopy and physical measurements were completed using stereomicroscopy and polarized light microscopy. The results are as follows: Microchemical solubility tests

Table 5. Summary table of optical microscopy and physical measurements for the PAINT samples.

Item #	Color/Layer Sequence	Thickness (um)	Image	Observations
Item 1 (Vehicle 1)	1. Clearcoat	40		1. Uniform, smooth
	2. Forest green with gold flakes	30		2. Coarse, flake
	3. White-gray primer	30		3. Uniform, smooth
	4. Medium gray electro-coat	5		4. Uniform, thin
Item 2 (Vehicle 2)	1. Clearcoat	38		1. Uniform, smooth
	2. Forest green with gold flakes	30		2. Coarse, flake
	3. White-gray primer	29		3. Uniform, smooth
	4. Medium gray electro-coat	6		4. Uniform, thin
Item Q3 (Victim)	1. Clearcoat	41		1. Uniform, smooth
	2. Forest green with gold flakes	30		2. Coarse, flake
	3. White-gray primer	30		3. Uniform, smooth
	4. Medium gray electro-coat	5		4. Uniform, thin

were also conducted. The results are given in Table 6.

Elemental analysis was conducted on each layer of the paint samples beginning with the clear-coat using μ -FTIR and SEM. The following images show the overlaid IR spectral comparison (Figure 22 – Figure 26)and the summary table from SEM is given in Table 7:

Table 6. Summary table of microchemical test results.

Item	Acetone	Xylene	DPA	Enamel or Lacquer
Item 1 (vehicle 1)	Not soluble Mica flakes migrated to edges	Softened clear coat	No reaction Minor bubbles	All layers enamel
Item 2 (vehicle 2)	Not soluble	Color coat softened and curled	No reaction	All layers enamel
Item 3 (victim)	Not soluble	Color coat softened and curled	No reaction	All layers enamel

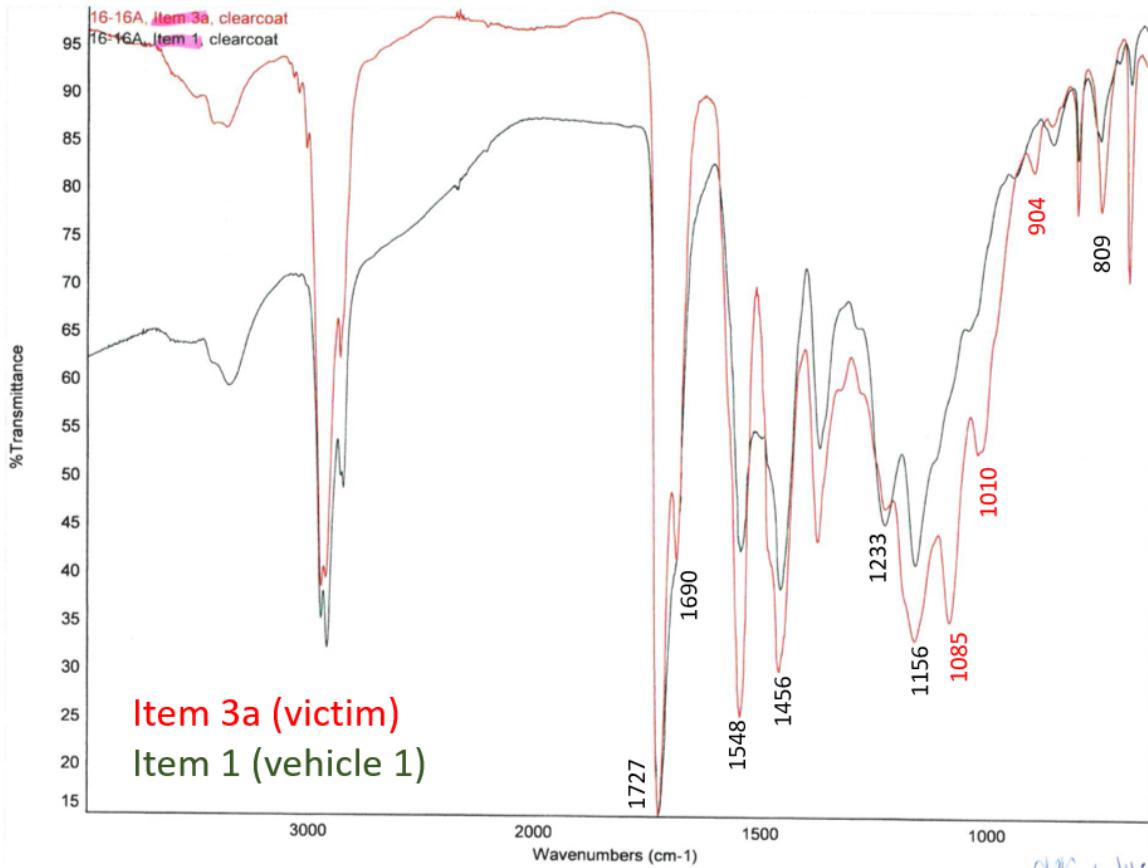


Fig. 22. Overlaid spectral comparison between the clearcoat from *Item 1 (vehicle 1)* and *Item 3 (victim)*.

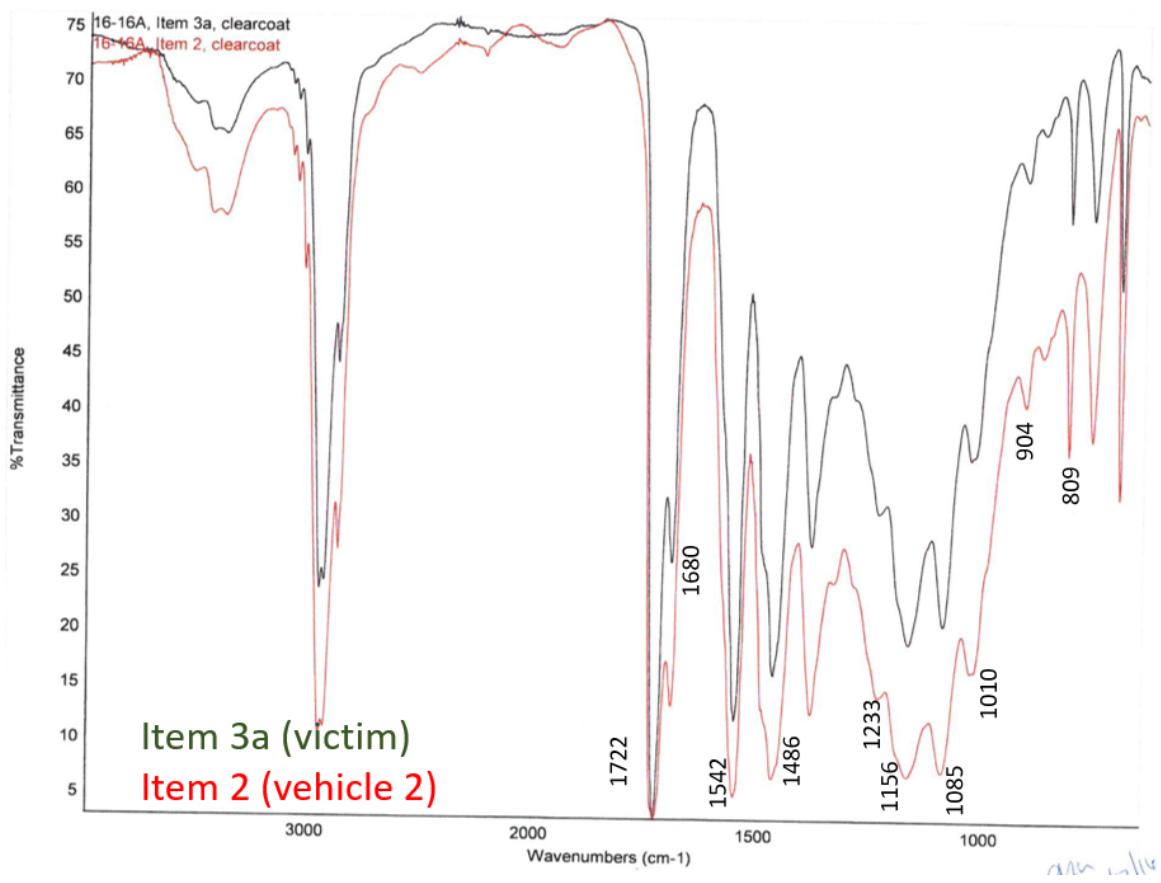


Fig. 23. Overlaid spectral comparison between the clearcoat from *Item 2 (vehicle 2)* and *Item 3 (victim)*.

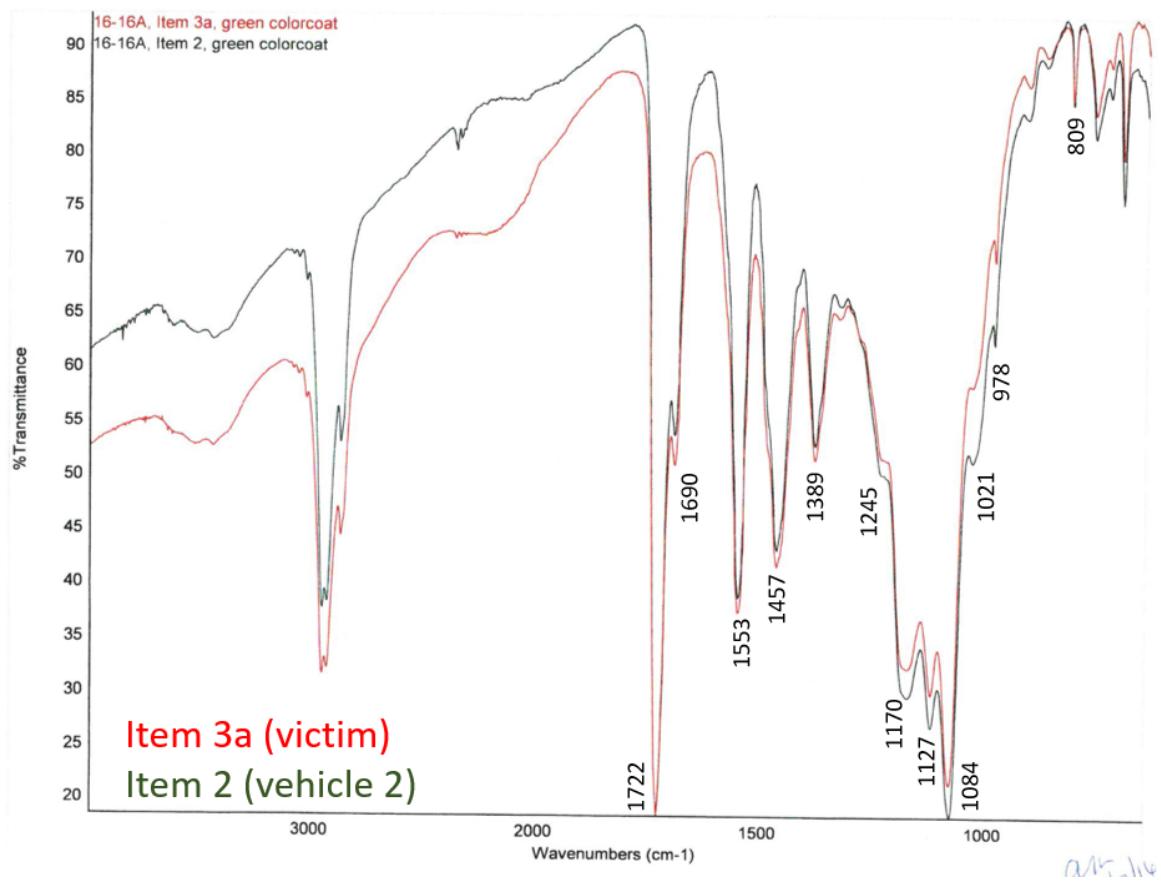


Fig. 24. Overlaid spectral comparison between the basecoat/color layer from *Item 2 (vehicle 2)* and *Item 3 (victim)*.

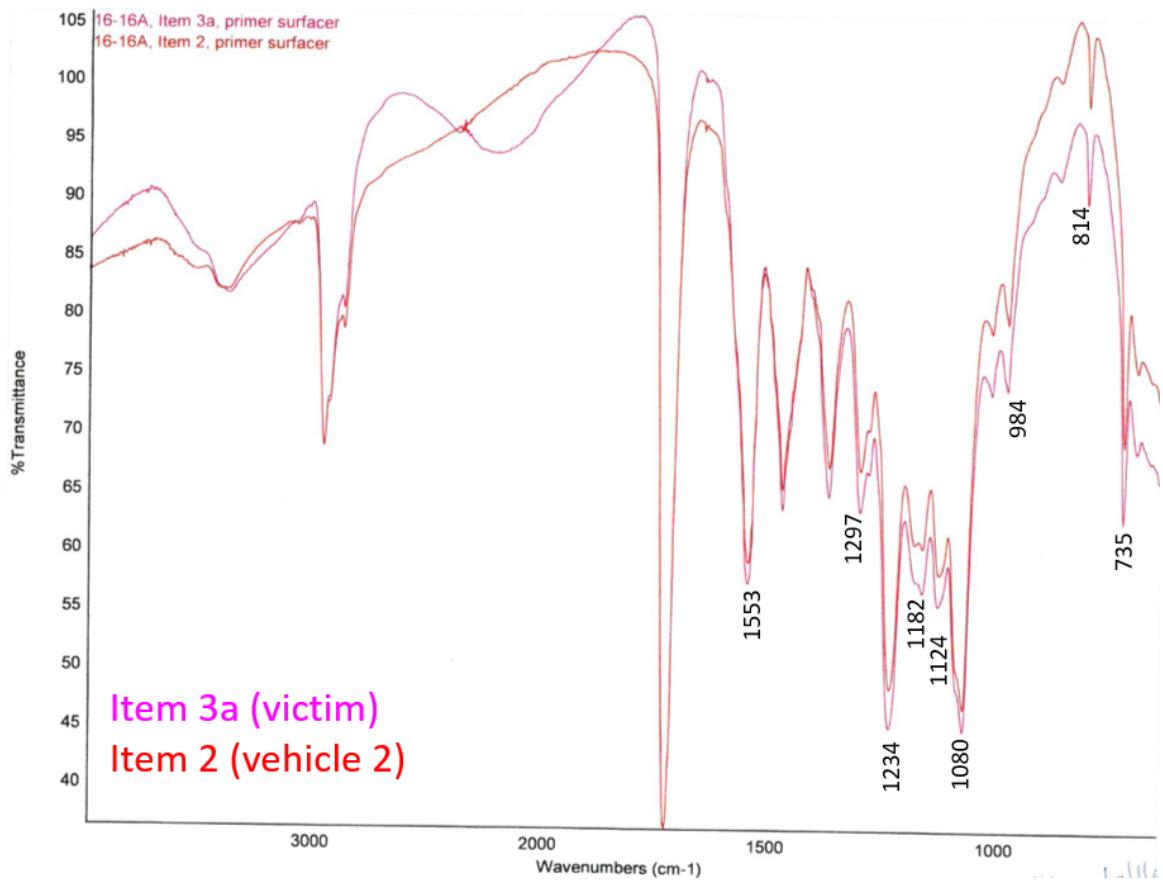


Fig. 25. Overlaid spectral comparison between the primer layer from *Item 2 (vehicle 2)* and *Item 3 (victim)*.

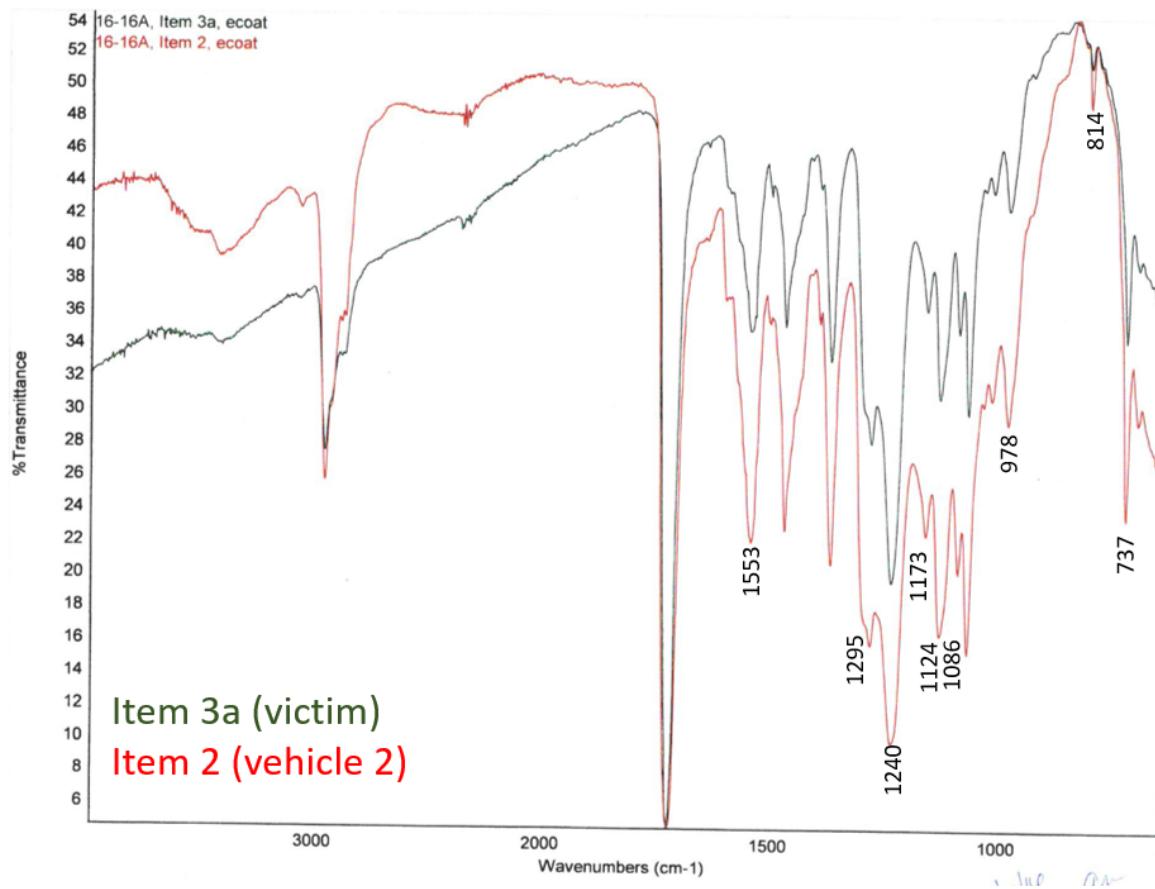


Fig. 26. Overlaid spectral comparison between the electro-coat base layer from *Item 2 (vehicle 2)* and *Item 3 (victim)*.

Table 7. Summary table of elemental analysis using SEM.

ITEM 2 (vehicle 2)	Major	Minor	Trace
Clear Coat, Embedded	Si	S	Al, Ba
Clear Coat, Peels	Si	S	
Color Coat, Embedded	Ti, Ba, S, Si, Al, Br	K, Cl	Fe, Cu
Color Coat, Peels	Ti, Ba, S, Si, Al, Br		Cu, Cl, K
Light gray primer, Embedded	Ba, Ti, S	Si	Al, Fe, Cl
Light gray primer, Peels	Ba, Ti, S	Si	Al, Fe, Cl, Ti
Medium gray primer, Embedded	Ba, Ti	Si, Al	Fe, S, Cl
Medium gray primer, Peels	Ti, S	Al	Fe, S, Cl

ITEM 3 (victim)	Major	Minor	Trace
Clear Coat, Embedded	Si	S	Al, Ba
Clear Coat, Peels	Si	S	
Color Coat, Embedded	Ti, Ba, S, Si, Al, Br	K, Cl	Fe, Cu
Color Coat, Peels	Ti, Ba, S, Si, Al, Br		Cu, Cl, K
Light gray primer, Embedded	Ba, Ti, S	Si	Al, Fe, Cl
Light gray primer, Peels	Ba, Ti, S	Si	Al, Fe, Cl, Ti
Medium gray primer, Embedded	Ba, Ti	Si, Al	Fe, S, Cl
Medium gray primer, Peels	Ti, S	Al	Fe, S, Cl

Glass Analytical Results:

After search of the PAINT chip samples (*Item Q3a & Item Q3b*) through the PDQ database, known glass samples were recovered from the Green Jeep Cherokee Trail Hawk 2015 – Vehicle 2:

- **K1 inner** – glass samples from inner side of vehicle 2 windshield
- **K1 outer** – glass samples from outer side of vehicle 2 windshield
- **Q3-1, Q3-2, Q3-3** – glass samples from sweater pocket of the suspect (vehicle 2 owner)

Optical microscopy and physical measurements were completed using stereomicroscopy and polarized light microscopy. All fragments/samples were found to be isotropic and clear. The results are as follows:

Table 8. Summary table of optical microscopy and physical measurements for the known GLASS samples (vehicle 2).

K1 Outer (vehicle 2)					K1 Inner (vehicle 2)				
Sample	Thickness (mm)	Dimensions (mm)	Type	Color	Sample	Thickness (mm)	Dimensions (mm)	Type	Color
K1O-1	4.36	5x4	Float	Clear	K1I-1	4.33	5x4	Float	Clear
K1O-2	4.36	5x3	Float	Clear	K1I-2	4.36	4x3	Float	Clear
K1O-3	4.4	10x7	Float	Clear	K1I-3	4.4	3x2	Float	Clear
K1O-4	4.37	10x5	Float	Clear	K1I-4	4.4	6x4	Float	Clear
K1O-5	4.4	5x4	Float	Clear	K1I-5	4.36	6x4	Float	Clear
K1O-6	4.37	4x3	Float	Clear	K1I-6	4.4	5x6	Float	Clear
K1O-7	4.4	3x7	Float	Clear	K1I-7	4.38	6x3	Float	Clear
K1O-8	4.36	12x5	Float	Clear	K1I-8	4.4	7x5	Float	Clear
K1O-9	4.4	5x6	Float	Clear	K1I-9	4.4	5x5	Float	Clear
K1O-10	4.38	6x3	Float	Clear	K1I-10	4.36	5x4	Float	Clear

Table 9. Summary table of optical microscopy and physical measurements for the questioned GLASS samples.

Questioned 1 (road)					Questioned 2 (victim)				
Sample	Thickness (mm)	Dimensions (mm)	Type	Color	Sample	Thickness (mm)	Dimensions (mm)	Type	Color
Q1-1	N/A	4x3x1	N/A	Clear	Q2-1	N/A	N/A	N/A	Clear
Q1-2	N/A	2x1x1	N/A	Clear	Q2-2	N/A	N/A	N/A	Clear
Q1-3	N/A	0.5x2x1	N/A	Clear					
Q1-4	N/A	0.3x1x1	N/A	Clear					
Q1-5	N/A	1x2x1	N/A	Clear					
Q1-6	N/A	1x2x1	N/A	Clear					
Q1-7	N/A	1x1x1	N/A	Clear					
Q1-8	N/A	1x0.5x0.3	N/A	Clear					
Q1-9	N/A	1x0.3x0.3	N/A	Clear					
Q1-10	N/A	0.5x3x4	N/A	Clear					

Questioned 3 (suspect)				
Q3-1	N/A	1x0.5x0.3	N/A	Clear
Q3-2	N/A	1x0.4x0.2	N/A	Clear
Q3-3	N/A	1.1x0.3x0.3	N/A	Clear

The refractive index was measured using the Foster and Freeman GRIM 3. The mean refractive index ($n=5$) of each fragment is given in Table 10 and is plotted in Figure 27.

Table 10. Summary table of refractive indexes for the known and questioned glass fragments.

Fragment	Mean RI (N=10)
K1 inner	1.5228470
K1 outer	1.5170410
Q1-1	1.5228400
Q1-2	1.5170525
Q1-3	1.5170450
Q1-4	1.5228175
Q1-5	1.5170250
Q1-6	1.5228500
Q1-7	1.5170250
Q1-8	1.5170500
Q1-9	1.5170400
Q1-10	1.5228300
Q2-a	1.5228275
Q2-b	1.5170350
Q3-a	1.5228350
Q3-b	1.5228275
Q3-c	1.5228350

Additionally, elemental analysis was conducted on the glass fragments using LIBS. The ratios of elements in the windshield from Vehicle 2 are presented in Table 11. Additionally, the average ratios of elements for $Q1a$, $Q1b$, $Q2a$, and $Q2b$ are given in Table 12 and the ratios of elements recovered from the sweater of the suspect ($Q3$) are given in Table 13. Note that with LIBS, three standard deviations from known dictate discrimination.

Notebook Requirement for the Lab

There is no lab notebook requirement for this lab. You will need to submit a forensic report for the lab. Be sure to include all piece of evidence, the analytical methodology applied (as if you performed the instrumental analysis), and an appropriate interpretation of the results.

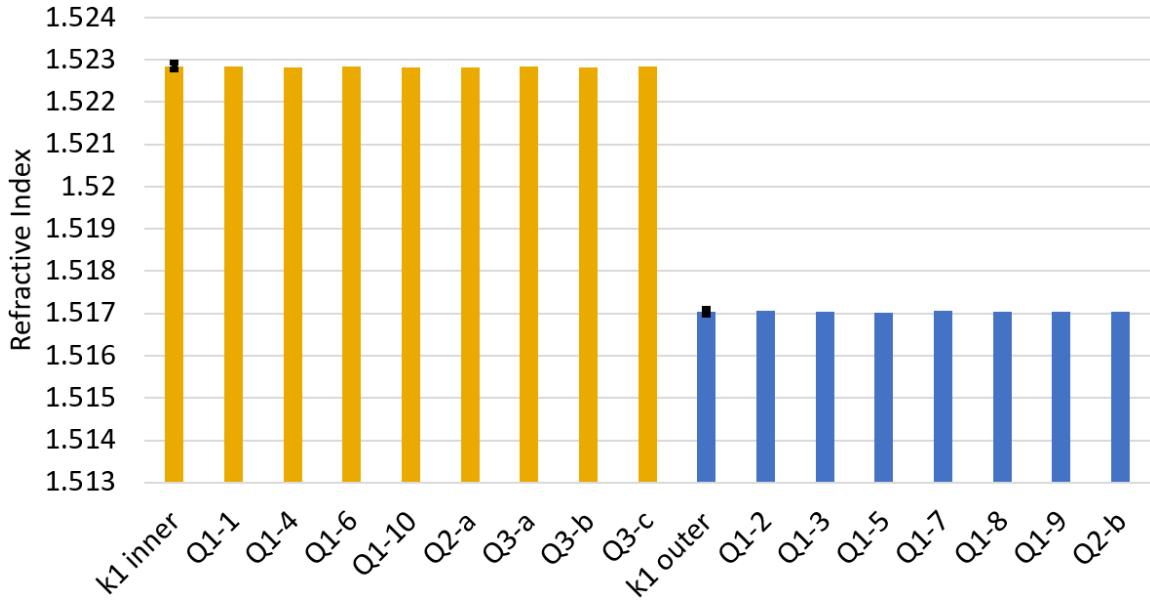


Fig. 27. Mean refractive index of each glass fragment. Note: do not use this figure to draw conclusions due to the size of the error bars, rather as guidance for evaluating the data in Table 10.

Table 11. Measured element ratios for the *K1* known glass fragments using LIBS.

	K1 windshield from vehicle 2 Jeep Cherokee Trail Hawk						
	Si288/Mg285	Ca 422/Na330	Al 396/Ca370	Sr407/Fe358	Ba454/Ca 422	Fe358/Ti336	Na819/K766
K1 inner 1-1	1.980939564	5.957119946	0.473437864	0.810277657	2.018118714	1.190446115	9.849379035
K1 inner 1-2	1.879028135	6.150063765	0.450437296	0.872796277	1.290260676	1.179190862	11.532472346
K1 inner 1-3	1.930315526	5.692396643	0.478177729	0.815872477	1.444466660	1.134842320	9.894719522
K1 inner 1-4	2.000895351	5.929323102	0.478247079	0.794710914	1.778717734	1.150098958	9.164804771
K1 inner 2-1	1.982496229	7.248655386	0.425751460	0.794455290	2.820989487	1.368833659	11.121826896
K1 inner 2-2	2.037461801	6.251344548	0.455060229	0.712959484	2.362080096	1.350037250	10.752803539
K1 inner 2-3	2.002723139	6.237964993	0.472677090	0.660247598	2.444084087	1.381931605	9.802414247
K1 inner 2-4	1.989241687	6.709031405	0.430615996	0.760243785	2.979118837	1.323511924	12.235988076
K1 inner 3-1	1.901234992	7.695578753	0.404420426	1.042908706	2.825628267	1.221546828	15.539763785
K1 inner 3-2	1.894444564	8.261773401	0.369033037	1.231200364	3.377281234	1.070011105	11.112758289
K1 inner 3-3	1.900150946	7.812250248	0.382506091	1.114733471	2.636547056	1.085817540	11.921373581
K1 inner 3-4	1.915270096	7.349412664	0.417699067	0.965818018	2.719196084	1.216696653	12.907817101
K1 outer 1-1	1.822335713	6.889331831	0.840766920	1.185764264	0.029898549	1.080318638	1.608994248
K1 outer 1-2	1.826632929	7.095885198	0.698489337	1.420846987	0.037596517	0.998477991	1.810261588
K1 outer 1-3	1.867789077	5.178191676	0.935976213	1.002739090	0.033829868	1.134078564	1.818647278
K1 outer 1-4	1.805624664	7.622916922	0.666653073	1.445502572	0.036514309	0.942926473	1.825130912
K1 outer 2-1	1.826497962	7.131138961	0.821627565	1.214081820	0.029674284	1.102709330	1.645260793
K1 outer 2-2	1.798726595	6.976818366	0.843244283	1.201811053	0.028504210	1.091311769	1.589481722
K1 outer 2-3	1.831615274	7.674019970	0.664835990	1.404547829	0.035917976	0.979130409	1.773448429
K1 outer 2-4	1.854959456	7.800339843	0.643477760	1.462625121	0.034651359	0.952154749	1.681826251
K1 outer 3-1	1.833072800	7.486305925	0.674462523	1.487558105	0.034945173	0.983280925	1.749081554
K1 outer 3-2	1.810439770	7.547114500	0.683418483	1.451251581	0.035752812	1.004541771	1.786842512
K1 outer 3-3	1.802131147	7.460005480	0.682300069	1.415601944	0.035389080	1.009392983	1.894314864
K1 outer 3-4	1.822608606	7.778106856	0.693639665	1.370778370	0.032898869	1.047797499	1.734068709

Table 12. Measured element ratios (n=10 per sample) for the *Q1* and *Q2* glass fragments using LIBS.

		Si288/Mg285	Ca 422/Na330	Al 396/Ca370	Sr407/Fe358	Ba454 /Ca 422	Fe358/Ti336	Na819/K766
Q1a	Mean	1.951791333	6.905939768	0.436402764	0.845577570	2.588234567	1.282251805	11.985975792
	SD	0.056395813	0.664718456	0.024374601	0.140553200	0.253975853	0.096039344	1.899040843
Q1b	Mean	1.778944841	7.473817997	0.742697614	0.910244678	0.030986727	1.068825948	1.618675746
	SD	0.122708869	0.242234645	0.023936335	0.108319620	0.012248846	0.029646438	0.240367930
Q2a	Mean	1.920917261	6.723479925	0.432112092	0.794089893	2.537562067	1.277632083	11.639209172
	SD	0.038981322	0.684061755	0.021330137	0.170833509	0.244698075	0.102872983	1.033217588
Q2b	Mean	1.795917261	7.335602852	0.739617793	0.972065246	0.036480091	1.061129358	1.639209172
	SD	0.089032908	0.106527602	0.015113419	0.025543520	0.007917210	0.034991623	0.128470586

Table 13. Measured element ratios for the *Q3* glass fragments using LIBS.

	Q3 (Glass from Suspect Sweater)						
	Si288/Mg285	Ca 422/Na330	Al 396/Ca370	Sr407/Fe358	Ba454 /Ca 422	Fe358/Ti336	Na819/K766
Q3c-1	1.823193554	6.513445477	0.483684834	0.812959484	2.680095823	1.337249503	11.613513458
Q3c-2	1.739676574	6.879649925	0.425941567	0.759841956	2.840866616	1.393160544	12.594383972
Q3c-3	1.994048991	6.709031405	0.443266944	0.724378505	2.444084087	1.650664706	11.553904968
Q3c-4	1.915771080	6.955787533	0.404246039	0.870000000	2.200000000	1.205283025	10.725013900