

Washington State Patrol



Crime Laboratory Division

Materials Analysis General Chemical Analysis Training Manual

June 2025

MA Gen Chem Analysis Training Manual	Page 1 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

TABLE OF CONTENTS

1	INT	RODUCTION	3
	1.1	Purpose and Scope	3
	1.2	EXPECTATIONS	3
	1.3	ORGANIZATION OF THE TRAINING MANUAL	4
	1.4	SAFETY	
2	CAS	SE AND ANALYTICAL APPROACH	5
	2.1	OBJECTIVES	5
	2.2	TOPIC AREAS	5
	2.2.	1 Screening Methods	6
	2.2.	2 Targeted Testing Methods	7
	2.3	READING AND REFERENCES	8
	2.4	STUDY QUESTIONS	10
	2.5	PRACTICAL EXERCISES	10
3	EVI	DENTIARY SIGNIFICANCE AND MOCK CASEWORK	12
	3.1	OBJECTIVE	12
	3.2	TOPIC AREAS	12
	3.3	READING AND REFERENCES	12
	3.4	STUDY QUESTIONS	12
	3.5	PRACTICAL EXERCISES	13
4	API	PENDIX A	14
	4.1	OC / PEPPER SPRAY	14
	4.2	BANK DYE	14
	4.3	ETHYLENE GLYCOL	14
G	ENER	AL CHEMICAL ANALYSIS TRAINING CHECKLIST	16

MA Gen Chem Analysis Training Manual	Page 2 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

1 INTRODUCTION

1.1 PURPOSE AND SCOPE

This manual contains an outline for training and/or assessing a forensic scientist in the area of General Chemical Analysis (GCA). The various study segments should be covered in the order presented.

This manual endeavors to promote and maintain consistency and quality among forensic scientists performing general chemical analyses across the Crime Laboratory Division. Certain inherent aspects of GCA prohibit the establishment of a rigid set of standard procedures to cover every case. Sufficient latitude should be given to allow for independent thought and individual freedom in selecting alternative courses of action. Upon completion of this training program, the trainee shall be thoroughly familiar with the options available to perform an examination of the most common types of evidence received.

1.2 **EXPECTATIONS**

The trainee is expected to have successfully completed the Primary Foundation Manual Module 1 and the following study segments from the Primary and Secondary Foundation Manuals: Thin Layer Chromatography, Gas Chromatography/Flame Ionization detector, Mass Spectrometry & Pyrolysis, Capillary Electrophoresis, Infrared Spectroscopy, High Performance Liquid Chromatography, Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy, X-Ray Fluorescence, Basic Practical Microscopy, Imaging and Visualization, Evidence Recovery, Special Applications in Microscopy, Volatile Extraction Techniques, Microchemical Methods, and Evidence Screening and Evaluation of Trace Evidence.

Trainees who have prior related training and experience can progress through the training program at an accelerated pace or skip certain study segments. The required documentation of such related training and/or experience shall be left to the supervisor in coordination with the technical lead(s) or their designee.

The instructor shall be experienced in the area of GCA. The instructor's casework and courtroom experiences, both prior and present, provide a unique aspect to the trainee's learning process that is impossible to duplicate in this training program. The instructor shall share such experiences with the trainee. The instructor shall also discuss with the trainee the training and reference materials (if any) available on the FLSB Portal. Although the trainee's primary interaction shall be with the assigned instructor, this program promotes and encourages discussions with other experienced examiners. When possible, the trainee should also take outside courses related to GCA.

The trainee shall maintain a notebook or multiple notebooks throughout the duration of this training program and shall record notes and observations for each study segment. The trainee notebook should be maintained in a neat and current fashion and should be present during conversations with the trainer. Upon completion of training, the trainee shall maintain the training notebook for the duration of their career. The form of the notebook(s) can be written, electronic, or a combination thereof.

The trainee is continuously evaluated throughout the training for comprehension and competency in theoretical knowledge, basic practical skills, and critical thinking skills. Training is progressive and continuously builds on and reinforces prior learning. Deficiencies on any of the training steps during the course of the training shall be rectified. It is important that these deficiencies be openly and promptly discussed among the trainee, trainer, technical lead, and/or supervisor, as appropriate. If necessary, training steps and testing can be repeated to satisfactorily complete this training program.

In order to successfully complete this training program, the trainee shall, after completion of all topic areas, successfully complete a closed book written exam passed with 80%, a competency exam passed with a 100%, and an oral testimony exam with a pass/fail. The completion of these steps shall be documented on a training checklist located at the end of this manual. The competency exam shall take the form of a mock case, which shall include a draft report. The oral testimony exam can either be a full

MA Gen Chem Analysis Training Manual	Page 3 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

moot court or an oral examination of testimony type questions between the trainer and the trainee. Supervised casework is optional and dependent on the trainee's repertoire of subdisciplines as well as performance on mock casework.

The trainer is responsible for preparing a request for authorization to be submitted through the trainee's chain of command when the trainee has successfully completed the GCA Training Manual. Training records, including records of training completion and authorizations, will be maintained in accordance with QOM requirements. Individual scientists are strongly encouraged to maintain copies of their own training records and their training notebook(s).

1.3 ORGANIZATION OF THE TRAINING MANUAL

This training manual consists of two segments, one covering the general analytical aspects of this type of analysis and the other covering the general casework aspects of this type of analysis.

Each study segment is comprised of five sections:

Objectives - Summarize the purpose of each study segment.

Topic Areas – Designates topics to be included in the study segment.

Readings – Lists the reference materials that should be read to complete the study segment.

Study Questions – Lists questions that assist the trainee in comprehension of the readings, promotes active discussion between the trainer and trainee, and documents understanding of the topic areas. Written answers to these questions shall be maintained in the training notebook as documentation of training.

Practical Exercises – Hands-on activities that are designed to provide the trainee first-hand experience with the main concepts of each study segment. Data or written explanation for each exercise shall be maintained in the training notebooks.

1.4 **SAFETY**

It is imperative that analysts take appropriate precautions during the analysis of general unknown evidence to handle these materials safely. Evidence from these cases can contain unknown materials that can present flammable, contact, and/or inhalation hazards in addition to any toxic effects. Special attention to the possibility of biological, nerve, or other toxins should be considered. Acids and bases can be encountered as evidence. These are very corrosive, and eye and skin protection shall be used. In addition, acids can be very reactive with chlorates, acetone, flammable liquids and water. Extreme care shall be taken when mixing these compounds.

Tear gas products are irritants, by definition, and can cause physical discomfort if inhaled. If working with spray products or clothing items containing high concentrations of tear gas products, perform analysis in a fume hood and avoid contact with skin and eyes.

Inadequately packaged samples need to be repackaged, and defective or damaged containers repaired or replaced.

As in all cases, care shall be taken to obtain as much advance information about the submitted item(s) as possible. The most useful information can often come from case investigators and their scene reports. Medical information regarding victim symptoms can be valuable, but it might be difficult or impractical to obtain due to HIPAA and confidentiality issues.

Due to the hazardous nature of many samples, it is recommended that scientists decline if possible to open evidence containers in a court of law or other public environment without thoroughly explaining the possible risks involved and the protective measures required before proceeding.

MA Gen Chem Analysis Training Manual	Page 4 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

2 CASE AND ANALYTICAL APPROACH

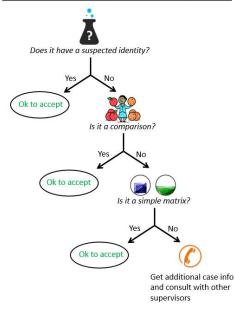
2.1 **OBJECTIVES**

- To familiarize the analyst with case approach for cases involving general chemical analysis of unknown substances.
- To familiarize the analyst with the case approach for cases involving suspected tampering.
- To learn to assess the potential hazard of a substance.
- To learn to evaluate the limitations inherent to various sample matrices.
- To learn to assess limitations due to packaging and storage conditions.

2.2 TOPIC AREAS

The Crime Lab has a limited capability to handle general chemical analysis cases. The Crime Lab's ability to provide useful forensic information needs to be evaluated before accepting this type of case. Cases where the Crime Lab cannot provide useful forensic information should not be accepted. Supervisors use a flow chart to help them determine if the Crime Lab has a reasonable chance of providing useful information and can be accepted.

Unknowns Analysis Case Acceptance



Before starting analysis on a general chemical analysis case, the scientist should also evaluate the case and the following limitations should be assessed:

- Can a suspected adulterant or substance be identified?
- Can the significance of identifying an adulterant or substance be determined?
- Can a fatal dosage of the unknown fall well below the detection limits?
- Has the evidence been appropriately packaged and stored to preserve its integrity (e.g. freeze evidence that may be at risk for decomposition)?
- Are there toxicology results and/or a Medical Examiner report?

General Chemical Analysis (GCA) cases can be separated into three categories:

- Target analyte where a known/specific compound is being identified,
- Adulterant/tampering where a known product is suspected of being altered, and
- Unknowns where the identity of the substance is not known.

MA Gen Chem Analysis Training Manual	Page 5 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

Although the evidence received in these cases can differ slightly, much of the analytical procedure is similar.

The submitting agency should be contacted and any available relevant information obtained regarding the circumstances of the case. Literature/internet research into the expected components of a material(s) suspected of having been adulterated and the suspected adulterant(s) is valuable in developing an appropriate analytical scheme, and in determining what the normal contents of the material(s) are. Safety Data Sheets and other manufacturer information can be valuable. In cases where the identity of the evidence is completely unknown, internet/literature research is usually conducted after preliminary analysis with screening tests has been performed.

Control samples of the untampered product(s) and suspected adulterant(s) are nearly always useful, if not essential in cases of suspected tampering. In general, the controls should be analyzed first to determine what components are present and verify that a proposed analytical scheme can be suitable.

Based on the case information obtained from the agency and research by the analyst, a particular substance or class of substances can become of interest. If so, analysis can proceed to targeted testing to determine the presence or absence of the particular substance(s) suspected.

In the instance that no single substance or class of substances is determined to be of particular initial interest, a general chemical screening process should be implemented to assess the presence of immediate hazards and narrow the scope of analysis. If at any point during analysis a particular substance or class of substances becomes of interest, analysis can proceed to targeted testing.

Occasionally it can be necessary to perform a cursory deformulation analysis to identify some or all of the components in an unknown in hopes of determining the identity of the submitted item.

If at any time, the analyst determines that the evidence is not suitable for the requested analysis, due to limited sample, spoilage, or other conditions, the analyst should discontinue the analysis. Analysis should also be discontinued if the analyst has exhausted all readily available techniques, even if no useful forensic information was obtained. If the evidence is determined to fit into a different service, the analyst shall write a report for the work performed and a new request will be opened for the appropriate analysis.

The analyst shall determine, in each case, which techniques and instruments to use and the order in which to conduct them. It can be useful to consult with other scientists before beginning analysis, especially if the sample is limited. Others can recognize an unknown material from its appearance which would significantly reduce its analysis time. The procedures chosen shall be based on the size of sample available weighed against the likelihood of obtaining useful information during each step of the analysis.

2.2.1 SCREENING METHODS

A visual examination of the evidence, often including a stereomicroscopic exam, should be conducted first, noting any obvious signs of tampering, the inclusion of extraneous material, or the absence of material. An assessment of the apparent homogeneity of the sample can be very important. Determine if any material suitable for direct analysis exists, such as tablet fragments, pockets of precipitate or foreign material, or sediments. Injection vials can be examined for broken seals and punctures of the septa.

For liquid samples, miscibility, pH, IR and GCMS analysis all can be useful to characterize the sample for identification or to aid in further extractions or analysis. Evaporation of a liquid to obtain solid residue which can be analyzed by IR, XRF, SEM/EDX, microscopy, or chromatography (after extraction with a suitable solvent) is often useful.

<u>Capillary electrophoresis (CE)</u> – CE as a screening test can be utilized to screen for or confirm anionic and cationic content, or to detect organic compounds including drugs. CE also can be useful in comparing

MA Gen Chem Analysis Training Manual	Page 6 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

questioned and known samples. Sometimes it is not possible to identify each component of the electropherogram, but comparing the overall pattern and ratios can still be informative.

<u>Gas Chromatography/Mass Spectrometry (GCMS)</u> – Due to the wide range of compounds that can be encountered in GCA casework, modified GC methods and/or MS parameters can be useful. Sample preparation procedures (such as dilution, dry extraction, acid/base extraction, derivatization, PAE, etc.) shall be appropriate for the analyte(s) in question. Before a neat sample is run on a GC, other tests should first be performed to ensure the sample doesn't damage the column or present other system complications. Multiple preparations and GCMS methods are sometimes necessary to characterize a sample as fully as possible. In some cases, pattern comparison of GCMS data between questioned and known samples is useful, or more useful than identification of specific compounds.

<u>High Pressure Liquid Chromatography (HPLC)</u> – HPLC is useful for samples that are not be volatile enough for GCMS analysis and are readily soluble in an appropriate solvent. Various columns can be selected to characterize organic, ionic, and polymeric materials. HPLC can prove valuable in comparing questioned and known samples by comparing the overall chromatographic pattern and ratios of peaks within that chromatogram. This method is most useful for Target Analyte cases.

Infrared Spectroscopy (IR) – Infrared spectroscopy more than any other instrumental technique is affected by the purity of the analyte. As such, the ability of IR to identify a poison or unknown material is likely to be confined to instances in which a relatively pure sample is isolated from its matrix. This can be done by picking discreet particles from the sample or by extraction. A vapor cell is useful for gases or the headspace above a liquid. IR is often useful in general characterization of the composition of a sample matrix. IR also can be used effectively in comparisons of a control or reference sample to questioned items, looking for differences between the samples; for this purpose, samples shall be prepared for IR analysis in a similar manner. For liquid samples it can be useful to obtain a background of the main component of the liquid prior to analyzing the sample.

<u>Microcrystal Tests</u> – There are numerous different microcrystal tests, several catalogued in various CLD manuals. They are often more useful for a targeted analysis to confirm the presence of anions, cations, or specific organic compounds.

Miscibility Testing – Can be useful when analyzing liquids.

<u>Refractive Index</u> – Refractive index testing can be helpful for some inorganic solid materials.

<u>pH Paper</u> – can be useful for liquids as information for identification or as a screening test before putting a sample on GCMS

<u>Polarized Light Microscopy (PLM)</u> – PLM can be used to characterize a sample and identify materials through refractive index determination or conoscopy.

<u>Spot tests</u> – Some chemical spot tests, including commercially available kits, are useful as sensitive screening tests for classes of analytes, while others are highly specific for a single substance.

X-ray Fluorescence Spectroscopy (XRF) and Scanning Electron Microscopy/Energy Dispersive Spectroscopy (SEM/EDX) – Generally, XRF is more useful for detection of heavier elements and for screening of samples while SEM/EDX is more useful for analysis of small particles and lighter elements. When elemental analysis rather than imaging is of primary concern, samples for SEM/EDX generally do not need to be coated. These techniques are of limited usefulness for liquids and shall be performed at ambient pressure.

2.2.2 TARGETED TESTING METHODS

Performing testing tailored to confirm the presence of a specific analyte(s) is warranted base on initial case information or on any positive results from screening tests. This can involve extractions designed to

MA Gen Chem Analysis Training Manual	Page 7 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

isolate substance(s) of interest, followed by tests in which specificity can be more a focus than sensitivity or broad response. By its nature, targeted testing requires that a specific analyte is tested for.

Some of the common target analytes received by the WSP Crime Laboratory include pepper (OC) spray, bank dyes and ethylene glycol spiked products; see appendix A for a typical approach to analysis.

Any of the techniques listed as screening tests can be employed as targeted tests if the indicated substance(s) is amenable to analysis by that technique.

Some chemical spot tests can be highly specific for a given analyte. The degree to which a spot test can be used as an identification method depends on this specificity. A combination of non-specific tests can, when used together, achieve a high degree of specificity for a substance. Literature supporting the spot test, analyte in question, and specificity should be documented in the casefile.

Any method published in a peer-reviewed scientific journal or academic textbook for the isolation or identification of a specific substance or class of substances shall be considered valid and appropriate for use provided that the method is successfully tested per QOM 19.3 with the relevant reference materials and blanks prior to application to case evidence.

Non-essential modifications to existing methods are acceptable, subject to the subsequent approval of the peer reviewer, and provided that standards, blanks and controls are successfully processed using all such modifications. Examples of non-essential modifications include but are not limited to scaling down extractions, the use of a longer or shorter GC column, lower initial temperature, higher final temperature, different solvent delay, different split ratio, longer temperature hold times, and higher/lower MS scan range.

Literature references for new methods shall be kept in the case file. The reference need not be cited in the Laboratory Report, but the basic technique(s) used should be listed.

It is not always necessary to analyze every item of evidence submitted. It is the responsibility of the analyst to perform sufficient testing to support any reported conclusions and to be responsive to the needs of the justice system.

The analyst shall use their best judgment to determine when enough work has been performed on a case, even if the identity of the material could not be determined. A complete identification of the material is not always possible.

The following is a list of resources that can be useful for certain General Chemical Analysis cases:

- Manufacturer websites
- Safety Data Sheets
- Wikipedia
- <u>Consumer Product Information Database</u> (formerly Household Products) supported by National Institute of Environmental Health Sciences (NIEHS) of the National Institutes of Health (NIH). https://www.whatsinproducts.com/pages/index/1
- McCrone's The Particle Atlas
- Chamot and Mason's Handbook of Chemical Microscopy
- Winchell's Optical Properties Books

2.3 READING AND REFERENCES

The listed references are not intended to be an exhaustive list. The trainer will make recommendations on which references will be used during the training.

Materials Analysis Technical (MATP) Procedures for Techniques, Instruments, and General Chemical Analysis

MA Gen Chem Analysis Training Manual	Page 8 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

Cavett, V., Waninger, E. M., Krutak, J. J., & Eckenrode, B. A. (2004). Visualization and LC/MS Analysis of Colorless Pepper Sprays?. *Journal of forensic sciences*, 49(3), JFS2003215-8.

Moffat, A. C., Osselton, M. D., Widdop, B., & Watts, J. *Clarke's analysis of drugs and poisons*. London: Pharmaceutical press. Any edition.

Darby, S. M., Miller, M. L., & Allen, R. O. (2001). Forensic determination of ricin and the alkaloid marker ricinine from castor bean extracts. *Journal of Forensic Sciences*, *46*(5), 1033-1042.

Dasenbrock, C. O., Ciolino, L. A., Hatfield, C. L., & Jackson, D. S. (2005). The determination of nicotine and sulfate in supermarket ground beef adulterated with black leaf 40. *Journal of forensic sciences*, *50*(5), JFS2005074-7.

Egan, J. M., Rickenbach, M., Mooney, K. E., Palenik, C. S., Golombeck, R., & Mueller, K. T. (2006). Bank Security Dye Packs: Synthesis, Isolation, and Characterization of Chlorinated Products of Bleached 1-(methylamino) anthraquinone. *Journal of forensic sciences*, *51*(6), 1276-1283.

Fung, T., Jeffery, W., & Beveridge, A. D. (1982). The identification of capsaicinoids in tear-gas spray. *Journal of forensic sciences*, *27*(4), 812-821.

Hennessy, S. A., Moane, S. M., & McDermott, S. D. (2004). The reactivity of gamma-hydroxybutyric acid (GHB) and gamma-butyrolactone (GBL) in alcoholic solutions. *Journal of forensic sciences*, *49*(6), JFS2003340-10.

HowStuffWorks.com. I've heard of bank robbers being foiled by a "dye pack" put in their money stash. What is a "dye pack"? Downloaded October 22, 2018 from https://science.howstuffworks.com/question671.htm

Hunsaker, D. M., Spiller, H. A., & Williams, D. (2005). Acute selenium poisoning: suicide by ingestion. *Journal of forensic sciences*, *50*(4), JFS2004247-5.

Jackson, D. S., Crockett, D. F., & Wolnik, K. A. (2006). The indirect detection of bleach (sodium hypochlorite) in beverages as evidence of product tampering. *Journal of forensic sciences*, *51*(4), 827-831.

Kurosaki, K., Nakamura, T., Mukai, T., & Endo, T. (1995). Unusual findings in a fatal case of poisoning with chromate compounds. *Forensic science international*, *75*(1), 57-65.

Lanaro, R., Costa, J. L., Cazenave, S. O., Zanolli-Filho, L. A., Tavares, M. F., & Chasin, A. A. (2015). Determination of herbicides paraquat, glyphosate, and aminomethylphosphonic acid in marijuana samples by capillary electrophoresis. *Journal of forensic sciences*, *60*, S241-S247.

Lewis, K., & Lewis, R. J. (2001). An assessment of four solvents for the recovery of 2-chlorobenzylidenemalononitrile and capsaicins from "CS" and "Pepper" type lachrymator sprays, and an examination of their persistence on cotton fabric. *Journal of Forensic Sciences*, *46*(2), 352-355.

Logan, B. (1993). Product tampering crime: A review. *Journal of Forensic Sciences*, 38(4), 918-927.

Marise, C., & Ross, P. (2011). Identification of 1-methylaminoanthraquinone on Australian polymeric bank notes. *Forensic science international*, 206(1-3), 207-210.

Papoutsis, I., Mendonis, M., Nikolaou, P., Athanaselis, S., Pistos, C., Maravelias, C., & Spiliopoulou, C. (2012). Development and validation of a simple GC–MS method for the simultaneous determination of 11 anticholinesterase pesticides in blood—clinical and forensic toxicology applications. *Journal of forensic sciences*, *57*(3), 806-812.

MA Gen Chem Analysis Training Manual	Page 9 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

Reynolds, P. C. (2008). Analysis of bank dye evidence and the challenges of Daubert hearings. *Forensic Science Communications*, *10*(1), NA-NA.Downloaded October 22, 2018 from https://archives.fbi.gov/archives/about-us/lab/forensic-science-communications/fsc/jan2008/research

Spicer Jr, O., & Almirall, J. R. (2005). Extraction of capsaicins in aerosol defense sprays from fabrics. *Talanta*, 67(2), 377-382.

Tarbah, F. A., Mahler, H., Temme, O., & Daldrup, T. (2001). An analytical method for the rapid screening of organophosphate pesticides in human biological samples and foodstuffs. *Forensic science international*, 121(1-2), 126-133.

Wilson, C. I., Ignacio, S. S., & Wilson, G. A. (2005). An unusual form of fatal ethanol intoxication. *Journal of forensic sciences*, *50*(3), JFS2004325.

2.4 STUDY QUESTIONS

- 1. Give at least four examples of signs that product tampering could have occurred.
- 2. Analysis of some mushrooms suspected of being laced with something indicates a low level of nicotine is present. What are the possible explanations for the results?
- 3. What ingredients can be found in sterile water?
- 4. What storage conditions could lead to destruction or degradation of evidence samples or evidence of tampering? Consider the physical state of the evidence as well as the material being submitted.
- 5. Discuss how you might know if you were working with a radioactive material, a liquid containing sarin, or a powder containing anthrax or ricin.
- 6. You are given a case containing an unknown powder. What techniques would you use to screen the substance?
- 7. You are given a case containing an unknown liquid. What techniques would you use to screen the liquid?
- 8. What methods and techniques are best for mixtures? Consider both solid mixtures and liquid mixtures.
- 9. What are the differences between identification, classification, categorization, and characterization?
- 10. What are the differences and similarities between a target analyte case, a product tampering case, and an unknown case?
- 11. What types of things are you looking for in a visual and stereomicroscope screening of evidence?
- 12. What is the chemical component of bank dye and what chemical is the target for analysis of bank dye?
- 13. What other materials contain the same dye as bank dye?
- 14. What is a lachrymator? Include common names and chemicals present in your answer.
- 15. Why does the technical/training manual not list a specific method or methods for analysis of pepper spray?
- 16. What are some common rat poisons, and can they be detected with the instruments available in our laboratory system?
- 17. What common products contain ethylene glycol?
- 18. Why is the "control" sample typically examined first in a suspected product tampering case?

2.5 PRACTICAL EXERCISES

The following practical exercises should be conducted and documented as if they are casework.

- Obtain a bottle of bleach and a drink (e.g. can of soda, can of energy drink). Remove and save control samples of the bleach and the drink. Then create a questioned sample by adding a measured amount of bleach to the drink (record the amount of bleach added in your notes). Analyze and compare the questioned sample to the control samples.
- 2. Obtain a bottle of nail polish remover (acetone, non-acetone, or both), and a bottle of orange juice. Remove and save control samples from each of the three bottles. Create a questioned

MA Gen Chem Analysis Training Manual	Page 10 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

- sample (or two) by mixing nail polish into orange juice. Analyze and compare the questioned sample(s) to the control samples.
- 3. Smear cheese on a solid surface (e.g. glass slide) and let stand overnight. Analyze the residue that remains.
- 4. Obtain an OC spray and a piece of fabric (e.g. shirt, jeans). Analyze the OC spray. Apply some of the OC spray to the fabric. Analyze a control region and an OC region of the fabric.
- 5. Obtain a sample of ethylene glycol and a can of cat food. Remove and save control samples of the ethylene glycol and the cat food. Then create a questioned sample by adding a measured amount of ethylene glycol to the cat food (record the amount of ethylene glycol added in your notes). Analyze and compare the questioned sample to the control samples.
- 6. Analyze a sample of bank dye on a typical surface/substrate.
- 7. Obtain an alcoholic beverage and a sedative (e.g. alprazolam). Remove and save control samples of the beverage and the sedative. Then create a questioned sample by adding a measured amount of sedative to the beverage (record the amount of sedative added in your notes). Analyze and compare the questioned sample to the control samples.
- 8. Obtain three GC vials from your instructor. Determine which vial(s) have puncture mark(s), documenting appropriately.
- 9. Analyze a set of biological materials by stereomicroscopy, PLM, and IR. The set should include, at a minimum, dried samples of blood, bone, fat, meat, and a green leaf.
- 10. Receive a sample set of unknowns prepared by your trainer. The sample set shall include at least 5 different samples. A sample can be heterogeneous or homogeneous. Analyze each sample and state a conclusion that answers the question, "What is it?"

MA Gen Chem Analysis Training Manual	Page 11 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

3 EVIDENTIARY SIGNIFICANCE AND MOCK CASEWORK

3.1 **OBJECTIVE**

- To familiarize the analyst with report writing for unknowns cases.
- To ensure appropriate documentation and report writing skills.
- To ensure appropriate techniques and confidence for court presentation.

3.2 TOPIC AREAS

Reporting should follow the CLD's Quality Operations Manual. The following guidelines are suggestions specific to GCA.

It is at the final discretion of the analyst and the peer reviewer to determine whether sufficient data has been collected to report the presence of any substance(s), and what if any qualifications or limitations should be included in the reporting of the substance.

Ambiguity or uncertainty in the identification of a substance need not preclude mention of that substance in the written report, but the reasons for any uncertainty shall be addressed.

The general type of material contained in the sample matrix should be listed in the report, for example, "a sample of acidic liquid," "a portion of a candy bar," or similar descriptive terms. More detailed components of the sample matrix (such as fat, sugar, vanilla, protein, and so on) need not be identified in the report but can be reported if determined to be relevant by the analyst.

In many cases, no adulterant is detected in a questioned sample, and unknown materials can remain unidentifiable. It is the scientist's responsibility and at their discretion to determine when all practical efforts have been made to characterize the evidence and to screen for any poisons or other likely components that might be present and detectable by the methods available. In general, sample size or condition and the laboratory's analytical capabilities are the limiting factors. In cases where no poison is detected or an unknown material remains unidentified, the Laboratory Report shall address the limitations of the Crime Laboratory to detect certain poisonous or harmful substances. Examples of limitation statements include, but are not limited to:

- This laboratory is not equipped to detect all possible toxic or harmful materials, especially in small amounts
- The Washington State Patrol Crime Laboratory does not have the capability to screen for most biological/protein or radiological hazards.
- Some harmful or toxic materials are not detectable by the methods available in this laboratory, especially biological or radiological materials, or substances present in small amounts.

If the evidence is found to be unsuitable for analysis, a report shall be prepared describing the reason analysis was discontinued or not performed.

3.3 **READING AND REFERENCES**

Materials Analysis Technical Procedures – General Chemical Analysis CLD Quality Operations Manual – Report Writing

3.4 STUDY QUESTIONS

- 1. How do you decide if a request to identify an unknown material qualifies as a GCA or a General Criminalistics type of case?
- 2. How do you decide if a request for product tampering qualifies as a GCA or a Seized Drugs type of case?
- Describe the process and decisions that need to be made to accept a request for GCA.

MA Gen Chem Analysis Training Manual	Page 12 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

- 4. An agency submits a request for analysis. The only item of evidence is a tuna casserole. The special instructions state "look for rat poison." Describe the conversation that you have with the agency as part of your assessment of the request.
- 5. Who decides what techniques and instruments to use and the order they are used?
- 6. Describe, in general, sources for methods not listed in the technical manual. Are these sources acceptable to use in casework? If so, what documentation is required in the case file?
- 7. When should an analyst discontinue their analysis?
- 8. What conclusions can be reached from a GCA case?
- 9. Can you conclude that an unknown sample is food? Biological? Zoological? Botanical?
- 10. What information should be included in your notes?
- 11. What information should be included in a report?
- 12. How does cognitive bias come in to play for these types of cases?

3.5 PRACTICAL EXERCISES

- 1. Review at least 5 GCA case files (including reports) and an additional 5 GCA reports. A representative file from each analyst should be included in the mix. The types of cases should be mixed (target analyte, adulterants, and unknowns, if possible). Consider requesting files from archives to review a sufficient number of case files.
- 2. Write a report for your findings in Chapter 2 practical exercises 1-5.
- 3. Work at least 6 GCA mock cases as if they were real cases. These cases should be realistic in the type of evidence submitted. The mock cases should include at least one target analyte case, one product adulteration case, and two unknown cases. Follow the requirements of the Technical Manual and include a draft report.
- 4. Perform at least 3 practice technical reviews. These reviews can be on copies of active GCA case files prior to the actual case files being technically reviewed by a qualified analyst or on mock GCA case files created for this exercise. If there are two or more trainees, they can cross technical review their mock cases.
- 5. Participate in an oral practice session to practice giving verbal answers to court type questions for GCA.
- 6. Discuss with other GCA scientists any court testimony experiences they have had.
- 7. Observe court testimony in GCA if possible.

MA Gen Chem Analysis Training Manual	Page 13 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

4 APPENDIX A

4.1 OC / PEPPER SPRAY

Many pepper spray products have an orange or reddish tint, which can aid in locating stains on clothing or other objects. If the staining is heavy enough it can even be apparent on black or red fabrics. If the staining is faint or colorless, difference in surface texture, reflection of light, or other aids can be used to locate material for analysis. Some pepper spray formulations include a fluorescent dye which can be seen with a UV lamp or ALS.

Helpful article for visualization: [Cavett, V., Waninger, E. M., Krutak, J. J., & Eckenrode, B. A. (2004). Visualization and LC/MS Analysis of Colorless Pepper Sprays?. *Journal of forensic sciences*, 49(3), JFS2003215-8..]

The principal components of OC spray are capsaicin and dihydrocapsaicin. Each of these is readily soluble in methanol, dichloromethane, and other solvents, and each is suitable for analysis by GC/MS. Once material for analysis is located, portions of the clothing or other substrate can be extracted or rinsed with the chosen solvent and the resulting solution analyzed by GC/MS. A typical GC/MS method used for most controlled substance cases can be useful to detect and separate capsaicin and dihydrocapsaicin.

Appropriate control samples should be analyzed and taken if possible, from unstained portions of the sample substrate.

4.2 BANK DYE

Bank dye packs consist of a small pyrotechnic device, essentially a smoke bomb, which can disperse a red staining dye once activated. The main component to be identified in bank dye residues is the red dye 1-methylaminoanthraquinone (MAAQ). Some bank dye packs can also contain the tear gas o-chlorobenzilidinemalononitrile (CS).

Samples of currency, fabrics, and other materials can be examined for the presence of MAAQ. The residue typically is red or pink depending on the amount of material present and the nature of the substrate on which it was deposited. If the stain is on a non-porous surface such as glass or hard plastic, it can be collected as powder for analysis by IR and GC/MS. If the stain is embedded, MAAQ can be extracted using methanol or dichloromethane and the solution analyzed.

If CS is present, it can be detectable by GC/MS. CS is soluble in methanol and dichloromethane and is coextracted with MAAQ by the above approach. Experience in the WSP Crime Lab had found it rare to find detectable CS in bank dye residues; it is not known if this is because CS was not included in the dye packs encountered, or if the CS is present in smaller quantities, or is less persistent than MAAQ. Similarly, signs of the pyrotechnic components of the dye packs have not been observed in the colored deposits from dye packs.

Appropriate control samples should be analyzed, taken if possible, from unstained portions of the sample substrate.

4.3 ETHYLENE GLYCOL

Adding an antifreeze to a water-based liquid lowers its freezing point and increases its boiling point. Ethylene glycol (EG) and propylene glycol are two automotive antifreeze additives. Ethylene glycol is the most common and is very toxic; propylene glycol is currently less common (although that could change) because it is a less toxic alternative. Antifreezes containing ethylene glycol have been used to poison beverages and food products and are frequently encountered in animal poisoning cases. They are said to have a sweet taste that attracts animals and children and are highly toxic.

MA Gen Chem Analysis Training Manual	Page 14 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

Often an item of canned pet food or meat is submitted soaked in antifreeze, which appears as a florescent green or pink liquid. As these items are typically stored in the vault freezer upon submission, it is telling when the item is removed from the freezer and has remained unfrozen despite being stored at freezing temperatures. Ethylene glycol is soluble in most organic solvents. A few drops of the liquid can be diluted in a milliliter of carbon disulfide or methylene chloride and run on a GC/MS method having a low oven starting temperature and scanning down to a low molecular weight. A typical fire debris method is ideal. In addition, EG is water soluble and can be examined using CE or HPLC.

MA Gen Chem Analysis Training Manual	Page 15 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1

GENERAL CHEMICAL ANALYSIS TRAINING CHECKLIST			
Trainee:	Trainer:	Trainer:	
	Trainee Initials/Date	Trainer Initials/Date	
Case and Analytical Approach			
Reading			
Study Questions			
Practical Exercises			
Evidentiary Significance & Mock Casework			
Reading			
Study Questions			
Practical Exercises			
Written Test			
Competency Exam			

MA Gen Chem Analysis Training Manual	Page 16 of 16	June 16, 2025
Approved by	All Printed Copies Are Uncontrolled	Revision 1