

**Title: General Approach to Chemistry Unit Operation (CHEM-01)****1. Purpose**

This procedure provides general guidelines for the analysis of chemistry-related cases within the Chemistry Unit (CU) of the Chemical Analysis Section (CAS) within the Division of Scientific Services (DSS) forensic laboratory. Within this document such things as types of cases, requests, sampling plans, and generalized workflows will be discussed.

**2. Scope**

This procedure describes general guidelines and information about evidence, case analysis requests, and reports related to items submitted to the Chemistry Unit of the DSS laboratory. These descriptions are based on the Laboratory's general laboratory (GL) procedures and summarize the overall approach to analytical practices within the Unit. Separate and detailed standard operating procedures (SOPs) exist that cover specific areas within each discipline.

**3. Responsibilities**

This document applies to unit personnel who contribute, or assist in contributing, results within a Chemistry Unit laboratory report.

**4. Specimens**

Types of evidence typically submitted to the Chemistry Unit for analysis involves items from fire investigations and shootings. While motor vehicle accidents, burglaries, and other cases that may involve paint evidence have been received in the past, due to low submission rates and other factors these cases are in the minority and are currently outsourced. Fire investigation-type cases usually involve evidence from fire scenes which are typically submitted within air-tight containers (e.g., unlined metal cans) due to the volatility and residual nature of possible ignitable liquids. Sometimes liquids can be submitted which may not necessarily need such cans. Comparison samples may be submitted. For crimes possibly involving firearms and the need to detect residues from ammunition primers, evidence may include, but not be limited to, gunshot residue (GSR, pGSR) stubs, GSR kits, and clothing.

**5. Standards and Controls**

- 5.1 Standards and controls should be listed within relevant procedures within the applicable discipline.
- 5.2 Materials will be purchased according to established laboratory and section practices and will be inventoried/checked upon receipt.
- 5.3 Critical reagents and reference standards will be verified prior to their use with casework. Such verifications typically involve using the applicable procedure with the materials and analyzing appropriately as is done with casework. Documents supporting such verifications should be kept with quality files.
- 5.4 Ignitable liquid commercial products will be verified as best as possible to their general classification. Because these are not certified reference materials, there usually are no certificates of analysis documents with these products. For those reference materials that will be used for ignitable liquid-type examinations, commercial product reference materials will be verified as to the ASTM Ignitable Liquid Classification Scheme (ASTM E1618 document).
- 5.5 Performance standard solutions or other QA/QC materials will be verified prior to use with instrument evaluations, whether for casework or otherwise. Documentation will be kept within appropriate files.
- 5.6 Any issues with purity or quality of materials during verifications will be cause to take such materials out of service and not be used with casework.
- 5.7 Storage of all materials and expiration dates will be according to manufacturer recommendation, if applicable. If no expiration date is available then the materials will be valid until one (1) year since date of last verification.

## **6. Calibration/Equipment/Maintenance of Equipment**

The verification of instrument calibration should be performed at regular intervals. The description of such verifications is beyond the scope of this procedure. Relevant equipment will be listed within applicable procedures within the associated discipline.

- 6.1 Instrument Operations
  - 6.1.1 Both preventive maintenance and routine quality assurance/quality control (QA/QC) checking will be performed.
  - 6.1.2 Specific instructions on such instrumental operation and QA/QC procedures should be found within applicable standard operating procedures (SOPs).
  - 6.1.3 Standards and/or controls will be analyzed using instrumentation to ensure the data and instrument sensitivity are adequate.

6.1.4 Before any instrument is used for casework its performance will be evaluated to ensure that accurate and reliable data will be generated from their use.

6.1.5 Validations will occur for instruments prior to their use with casework. Documentation will be kept in appropriate validation binders and approval for use will follow General Laboratory (GL) and Section policies.

## 6.2 Preventative Maintenance

6.2.1 Where applicable, service maintenance agreements from vendors have been established to help facilitate continual instrument operability.

6.2.2 Oil within vacuum rough pumps connected to instruments should be routinely checked for color and level. Change oil at suggested intervals (e.g., 6 months) or when deemed necessary.

6.2.3 If instrument problems persist or instruments do not pass QA/QC evaluations then signs indicating not to use will be placed on such instruments so other analysts know not to use them for casework.

6.2.4 Instruments will be evaluated prior to casework and should be evaluated whenever necessary (e.g., after power outages).

## 7. Sampling

Sampling should be described within relevant procedures within the applicable discipline.

## 8. Procedure

Provided below are procedures for the general operational flow and casework analysis within the Chemistry Unit.

### 8.1 Case Generation and Evidence Handling

8.1.1 Cases are usually created and requests are generated by Evidence Receiving Unit (ERU) personnel. Official notification to the appropriate analyst/Lead is usually done through the Laboratory Information Management System (LIMS) using JusticeTrax' (JT) LIMS-plus software. Additional or alternate notification can initially occur through other methods (e.g., telephonically, e-mail).

8.1.2 Evidence will be transferred to analysts according to general laboratory (GL) practices and is normally done through the Evidence Receiving Unit (ERU).

8.1.3 Analysts should retrieve evidence as soon as possible and should inventory in a timely manner (i.e., not longer than 30 days upon receipt).

- 8.1.4 Analysts will ensure that evidence is under proper seal (e.g., evidence tape and initials) before acceptance and/or when transferring.
- 8.1.5 If evidence is found to not be under proper seal during transfer then analysts may assist with such proper sealing prior to their acceptance of the evidence. They will ensure that such activities are recorded within relevant documents.
- 8.1.6 If evidence does not lend itself to being under proper seal (e.g., too bulky) then evidence may still be accepted by analysts but proper steps need to be taken to ensure evidence preservation and integrity. The appropriate Deputy Director, or their designee, will be notified when evidence can't be placed under proper seal or when other unusual activities arise.
- 8.1.7 All evidence shall be stored in an appropriate locked cabinet or designated evidence room and shall be stored under proper seal when not under active examination. A proper seal is defined as one which prevents evidentiary loss, cross-contamination, and un-noticed deleterious change. Active examination is defined as a time period wherein a single case is analyzed during a limited time period (e.g., the same day).
- 8.1.8 Even during active examination, whenever there is a possibility that evidence could be lost, contaminated, or unknowingly tampered then such evidence should be placed under proper seal. Examples of proper seal include, but are not limited to, using evidence tape with inked initials across tape edges or heat-seals with inked initials over seals.
- 8.1.9 For evidence which cannot fit into an appropriate evidence locker or evidence room, an appropriate Deputy Director or their designee will be notified and one or more of the following will occur:
- 8.1.9.1 An alternate evidence room will be selected from within the DSS laboratory (appropriate authorization needs to have been obtained if using rooms from other sections).
- 8.1.9.2 Evidence may be left outside an evidence locker or evidence room provided that the evidence is under proper seal and the environment where it is placed has limited access and is acceptable to store such evidence. Signs indicating that evidence is being stored within the area must be posted on every entrance to that area so employees know that unnecessary traffic in that area should be avoided.
- 8.1.9.3 Evidence not able to be placed under proper seal may be stored in a room/area for limited duration but signs should be placed on all entrances to the room/area indicating unsealed evidence is being stored (e.g., 'No Entrance – Examination in Progress'). Further steps should be taken to limit the access (e.g., temporarily changing proximity card reader authorization) so that only authorized employees are allowed in that area for that time period.

- 8.1.10 The transfer of evidence must be done using a chain of custody system.
- 8.1.11 Upon starting examinations, if possible, analysts should either try to preserve the original seals or photograph such seals prior to alteration. Accessioning areas, examination areas, and areas where evidence is re-sealed will be clean and in locations where no contamination will occur.
- 8.1.12 While under examination and in a secure environment (e.g., laboratory or instrument room) evidence does not have to be under proper seal (i.e., free from sample loss, free from contamination, and able to be detected if tampered).
- 8.1.13 Instances where evidence will be consumed during analysis and unavailable for future re-testing should be conveyed to appropriate customers prior to such analysis. Evidence which will be preserved but which may be in another state/form (e.g., extracts from debris within a can) and which will be sent back to the submitting agency should be treated as evidence (i.e., labeled, placed under proper seal, and tracked appropriately) according to laboratory/section policy.
- 8.1.14 After performing all requested examinations and analyses evidence will be placed under proper seal. If new packaging is needed then old packaging can be isolated and labeled as 'original packaging material.' Containers housing items will be marked so that items and other relevant case information can be easily identified.
- 8.1.15 Evidence is stored in appropriate secured evidence storage areas until returned to the Evidence Receiving Unit or transferred to another analyst.
- 8.1.16 Plastic tamper-proof evidence ties with analyst handwritten initials and dates can be used on an evidence storage container/locker to help ensure evidence integrity. Documentation of the use of such ties is not required.

Note: Evidence within an instrument or on autosampler/autoinjector devices need not be under proper seal nor their location tracked.

## 8.2 LIMS and Case Documentation

- 8.2.1 Evidence is tracked, casework documentation is maintained, and specific requests for disciplines are created through the use of a laboratory information management system (LIMS) called LIMS-plus.
- 8.2.2 Members of the Chemistry Unit will work with ERU and Case Management Unit (CMU) staff, as well as other laboratory staff, in order to coordinate evidence transfers, update information about cases, issue reports, and update other relevant aspects regarding cases (e.g., testimony, subpoenas).

- 8.2.3 The description/inventorying/accessioning of evidence, taking of case notes, and generation of other documentation will be the responsibility of the assigned analyst and will normally be captured within the case file. The term 'case file' is a combination of hardcopy documents within case file folders and electronic data within LIMS-plus.
- 8.2.4 All case documentation will be according to laboratory policy and relevant Unit SOPs.
- 8.2.5 Labels put on evidence and/or paperwork need only be accurate regarding case laboratory number, evidence item identification number, and general description. Evidence descriptions within LIMS-plus may change slightly based on the needs of the Laboratory or other sections. Printing/placing new labels is not necessary if such changes do not impact the use of the label in question.
- 8.2.6 All relevant communications, especially those with outside entities, must be captured within case files. All relevant e-mails should be captured and placed into case files. A suggested method for documenting such communications is to print e-mails into pdf-files, make their filenames into a sequential format according to case files (i.e., E-Mail-01 (MM-DD-YYYY)\_YY-1234), and record the e-mail within LIMS-plus' synopsis section with detailed information (e.g., date and analyst full last name). Other non-e-mail communications can be logged using the 'case activity' feature within LIMS-Plus.

### 8.3 Case Analyses

- 8.3.1 The analysis of evidence will be performed according to applicable SOPs within the Unit. Any minor deviations (i.e., those that don't affect the overall quality of analyses) will be documented and retained. Major deviations (i.e., those that may affect the overall quality of analyses) will be preapproved according to laboratory policy prior to occurrence.
- 8.3.2 Supervisors, Lead Examiners, and other analysts may all be utilized for assistance/discussions, where applicable and appropriate.
- 8.3.3 All appropriate documentation should be incorporated, where feasible, into the LIMS-Plus system. Supplemental documentation can be kept within hardcopy case file folders. Laboratory document control policies will be followed and all appropriate initials/dates will accompany relevant paperwork.

Note: Any additional stickers with initials (e.g., case file folders) should be appropriately filled out but are not required if not dictated within applicable Laboratory SOPs.

- 8.3.4 Once cases and requests are established within LIMS-plus they are assigned to appropriate analysts.

- 8.3.5 Evidence is transferred, analyzed, and results are obtained. All documentation, including photographs, will be maintained according to Laboratory policy. Instrumental data will be retained and stored according to both Laboratory and Section policies.

General Analytical Instrumentation used within the Chemistry Unit:

	pGSR	Ignitable Liquids / Residues
Scanning Electron Microscopy/ Energy Dispersive X-ray Spectroscopy (SEM/EDS)	x	
Gas Chromatography/Mass Spectrometry (GC/MS)		x
Headspace-Gas Chromatography/Mass Spectrometry (Flame Ionization Detection) (HS-GC/MS(FID))		x (possible)
Fourier-Transform Infrared (FTIR) Spectrophotometry (with Attenuated Total Reflectance (ATR) Accessory)		x (possible)

#### 8.4 Results and Reports

- 8.4.1 Analysts will summarize their results and include them within LIMS-plus to produce a 'draft completed' laboratory report.
- 8.4.2 Analysts will assign reviews within LIMS-plus (i.e., technical reviews (TR) and administrative reviews (AR)).
- 8.4.3 Reports will be appropriately reviewed and milestones will be captured. If any corrections to reports or the case file are required prior to signing-off on a review then the reason for the correction will be recorded within the 'Reviewer' text box in LIMS-plus (along with reviewer name and date).
- 8.4.4 Reports will be released and distributed to submitting agency representatives appropriately. If tracking of reports are necessary then that will be done within LIMS-plus as sub-itemized pieces of evidence.
- 8.4.5 All appropriate case documentation can be found either in physical case file folders or within appropriate cases in LIMS-plus. Analytical data must support the

results/conclusions within reports and a competent scientist should be able to come up with the same result/conclusion based on documents retained within case files.

- 8.4.6 Reports will be in the format according to Laboratory Policy and will generally be dictated by Crystal Report templates found within the LIMS environment.
- 8.4.7 All items that were received and analyzed will be listed within reports and appropriate comments/results about each will be described.
- 8.4.8 Supplemental reports are those which contain additional information to previous reports. Such reports must be able to be linked to previous reports for clarity of information.
- 8.4.9 Amended (or revised) reports are those which contain corrections to previous reports. Such reports must also be able to be linked to applicable previous reports for clarity.
- 8.5 Validations will occur for procedures prior to their use with casework. Documentation will be kept in appropriate validation binders and approval for use will follow General Laboratory (GL) and Section policies.

## **9. Decision Criteria, Calculations, Measurement Uncertainty**

Not applicable.

## **10. Abbreviations**

Below are some abbreviations which may be found within documents related to the Chemistry Unit. More abbreviations may be found within specific unit procedures.

- 10.1 GC/MS: gas chromatography/mass spectrometry
- 10.2 HS-GC/MS(FID): headspace-gas chromatography/mass spectrometry (flame ionization detection)
- 10.3 GC: gas chromatograph or gas chromatography
- 10.4 MS: mass spectrometer or mass spectrometry
- 10.5 MSD: mass selective detector
- 10.6 TIC: total ion chromatogram
- 10.7 SIM: selected ion monitoring
- 10.8 PIEI: positive ion electron impact
- 10.9 Char tube: charcoal tube adsorption/elution extraction technique
- 10.10 QA/QC: quality assurance/quality control



10.11 JT: JusticeTrax

10.12 FTIR-ATR: Fourier-transform infrared spectrophotometer with attenuated total reflectance

10.13 lpd: light petroleum distillate

10.14 mpd: medium petroleum distillate

10.15 hpd: heavy petroleum distillate

10.16 cs<sub>2</sub>: carbon disulfide

10.17 SEM: scanning electron microscopy

10.18 EDS: energy dispersive X-ray spectroscopy

10.19 GSR: gunshot residue (synonymous with pGSR)

10.20 pGSR: primer gunshot residue (synonymous with GSR)

10.21 AA: Atomic Absorption

10.22 keV: kilo-electron volt

10.23 cm<sup>-1</sup>: reciprocal centimeters

## **11. Limitations**

Limitations should be described within each Laboratory report along with any other information which may be deemed relevant to the submitting agency or reader of the report.

## **12. Safety**

Not applicable.

## **13. References**

DSS laboratory General Lab (GL) procedures

Chemical Analysis Section procedures

*Approved by Director: Dr. Guy Vallaro*

<b>Revision #</b>	<b>History</b>
1	New document. Takes the place of older SOPs.
2	Added clarification of how to verify commercial product reference materials in Section 5 ; Updated General Analytical Instrumentation used within the Chemistry Unit table ; removed numbers from 'Notes' ; Updated the definition section ; changed LIMS-Plus to LIMS-plus ; Minor formatting changes throughout document. Removed and/or updated information related to paint analysis.