



Laboratory Services Explosives

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2NDPA:	2-nitrodiphenylamine
4NDPA:	4-nitrodiphenylamine
AA:	ascorbic acid
ACN:	acetonitrile
AKII:	akardite II
AN:	ammonium nitrate
ANFO:	ammonium nitrate and fuel oil
ATR:	attenuated total reflectance (an FTIR technique)
AWG:	American wire gage
BA:	benzoic acid
BATS:	Bomb Arson Tracking System
BP:	black powder
CAN:	calcium ammonium nitrate
CCA:	copper-clad aluminum
CD:	conductivity detector
DAD:	diode array detector
DB:	double base; as in DBSP, double base smokeless powder
DBP:	dibutyl phthalate
DCDA:	dicyandiamide; also known as dicyanodiamide and cyanoguanidine
DCM:	dichloromethane, also known as methylene chloride
DEGDN:	diethylene glycol dinitrate
DEP:	diethyl phthalate
DI:	deionized
DMDNB:	2,3-dimethyl-2,3-dinitrobutane; also known as DMNB
DnPP:	di-n-pentylphthalate
DNT:	dinitrotoluene
DPA:	diphenylamine
DLaTGS:	deuterated L-alanine doped triglycine sulfate, a type of IR detector



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DTGS:	deuterated triglycine sulfate, a type of IR detector
EC:	ethyl centralite
EDS:	energy dispersive X-ray spectroscopy
EGDN:	ethylene glycol dinitrate
EIP:	extracted ion profile
ETN:	erythritol tetranitrate
EtOH:	ethanol
EVA:	ethylene vinyl acetate
Ex:	exhibit
FTIR:	Fourier transform infrared, a type of IR spectroscopy
GC:	gas chromatography
HE:	high explosive
HME:	homemade explosive
HMTD:	hexamethylene triperoxide diamine
HMX:	cyclotetramethylene tetranitramine (also octogen)
HPLC:	high performance liquid chromatography
HTGC:	high temperature gas chromatography
IC:	ion chromatography
IR:	infrared
IST:	ignition susceptibility test
LC:	liquid chromatography, see HPLC, UPLC
MC:	materials control (in context of sample preparation)
MC:	methyl centralite (in context of smokeless powder additives)
MCT:	mercury cadmium telluride, a type of IR detector
MeOH:	methanol
MS:	mass spectrometry
MTN:	metriol trinitrate
NC:	nitrocellulose



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NG:	nitroglycerin
NM:	nitromethane
PB:	post-blast
PDA:	photodiode array
PETN:	pentaerythritol tetranitrate
PLM:	polarized light microscopy
PTFE:	polytetrafluoroethylene
PVC:	polyvinyl chloride
PyGC:	pyrolysis gas chromatography
RDX:	cyclotrimethylene trinitramine (also hexogen)
RSP:	render safe procedure
SA:	Special Agent
SB:	single base; as in SBSP, single base smokeless powder
SEM:	scanning electron microscope
SP:	smokeless powder
TATP:	triacetone triperoxide
TIC:	total ion chromatogram
TNT:	trinitrotoluene
TPI:	threads per inch
UN:	urea nitrate
UPLC:	ultra performance liquid chromatography
UPW:	ultra-pure water, $\geq 18.2 \text{ M}\Omega$
UV:	ultraviolet
XRD:	X-ray diffraction, also known as X-ray powder diffraction or XRPD
XRF:	X-ray fluorescence spectroscopy



ATF-LS-E3 Scanning Electron Microscopy with Energy Dispersive Spectroscopy	ID: 796 Revision: 7
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1. Scope

- 1.1. Scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS) is a non-destructive analytical technique used to identify the elements present in materials. EDS is capable of detecting elements from boron (B) to uranium (U), at levels as low as 1% in a mixture.

2. Instrumentation/Reagents

- 2.1. Specifics regarding the SEM and EDS instrumentation (including instrument manufacturer, model, and software) and operating conditions shall be included on the data or on a separate parameter sheet that will be maintained in the case jacket.

3. Safety Considerations

- 3.1. No special precautions are required.

4. Procedure or Analysis

4.1. Sample Preparation

- 4.1.1. All samples should be secured to the stub with a conductive medium (carbon tape/tab, carbon paint, Duro-Tak, etc.), unless a beryllium planchet is used. Caution should be used if copper tape is used - refer to section 5.2.2 for considerations.
- 4.1.2. Nonconductive materials can be analyzed with the SEM in the low vacuum mode to reduce charging.
- 4.1.3. Powder samples must be adequately secured so that excess powder is not displaced when vacuum is applied to the sample chamber or during exposure to the electron beam.
- 4.1.4. Adjust the spot size accordingly to balance adequate signal and reduce charging.

5. Quality Assurance and Controls



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5.1. Calibration

- 5.1.1. Through regular calibration and upkeep of instrument logbooks, the quality of the EDS method is maintained.
- 5.1.2. The instrument will be calibrated within one month (up to 31 days) of the analysis. Additionally, the instrument should be calibrated after maintenance is performed on the SEM or EDS, including filament replacements for the SEM.
- 5.1.3. See instrument manual or work instructions for calibration instructions. The date of the calibration and the instrument-specific calibration parameters to be recorded shall be available in each instrument's calibration/maintenance logbook.
- 5.1.4. Refer to the EDS software and/or instrument manual(s) for the criteria that must be met for a successful calibration

5.2. Possible Sources of Error

- 5.2.1. The technique does have limitations. Since the transitions obtained are dependent upon the voltage (kV) used for the analysis, changing the applied voltage will result in a limitation on the transitions detected.
- 5.2.2. Copper tape should not be used if Cu is suspected in the sample, if the Cu energy lines would interfere with other elements (possibly Na), or if semi-quantification is being performed (e.g. for comparison purposes). Prior to associating Cu with a sample, that sample is required to be further analyzed using non-copper mounting material.
- 5.2.3. EDS counts may be low if the spot size is too small.
- 5.2.4. Extraneous peaks (e.g., sum and escape peaks) may become more prominent when the count rate is high.
- 5.2.5. Small Al peaks from the stub may be seen if the sample is close to the edge of the mounting tape.

6. References

- 6.1. Jenkins, R.; deVries, J.L., *Practical X Ray Spectrometry*, Springer Verlag, 1967.
- 6.2. Postek et al, *Scanning Electron Microscopy: A Student's Handbook*, 1980.



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- 6.3. *Birks, L.S., X Ray Spectrochemical Analysis, Interscience Publishers, 1959.*
- 6.4. *Smith, D.M., Visual Lines for Spectroscopic Analysis, Hilger and Watts, Ltd., 1952.*
- 6.5. *Jenkins, R. An Introduction to X Ray Spectrometry, Heyden and Son, Ltd., 1974.*
- 6.6. *Jenkins, R.; deVries, J.L., Worked Examples in X Ray Analysis, Macmillan Press Ltd., 1971.*
- 6.7. *Beveridge, A. (ed), Forensic Investigation of Explosions, published by Taylor & Francis Ltd., 1998.*



ATF-LS-E4 X-Ray Fluorescence Spectroscopy	ID: 784 Revision: 7
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1. Scope

- 1.1. X-ray fluorescence spectroscopy (XRF) is a non-destructive analytical technique used to identify elements present in solid, powdered or liquid samples. XRF is capable of detecting elements from sodium (Na) to uranium (U), at trace levels as low as 1% in a mixture.
- 1.2. The technique does have limitations. Since the transitions obtained are dependent upon the voltage (kV) used for the analysis, differing the applied voltage will result in a limitation to the transitions detected.

2. Instrumentation/Reagents

- 2.1. Specifics regarding the XRF instrumentation (including XRF manufacturer, model and software) and operating conditions shall be included on the data or on a separate parameter sheet that will be maintained in the case jacket.

3. Safety Considerations

- 3.1. Do not open sample chamber while x-rays are on (possible exposure to x-rays), unless the instrument is specifically designed to allow this.
- 3.2. Do not open or close chamber door rapidly, since sample may be lost.

4. Procedure or Analysis

- 4.1. Light, powdery samples must be secured if run under vacuum, so they are not lost/moved when vacuum is pulled.
- 4.2. Liquid samples should be examined under partial or no vacuum, so they do not boil off when vacuum is pulled.
- 4.3. Optimize the particle irradiation by adjusting the voltage, current, and the amp time as appropriate.

5. Quality Assurance and Controls



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5.1. Through regular calibration using known standards, and upkeep of instrument logbooks, the quality of the XRF method is maintained.

5.2. Control standards of mounting media (e.g. tape, Mylar film, etc.) should be run when necessary and maintained with the instrument or in the case jacket in order not to confuse background peaks with sample peaks.

5.3. Calibration

5.3.1. The instrument shall be calibrated within one month of the date used by using the aluminum/copper standard provided with the instrument. Additionally, the instrument shall be calibrated anytime a change in normal operations is detected.

5.3.2. New calibration values (including resolution, zero and gain), as well as the energy cut-off level (actual kV output), the date of the calibration, and user must be noted in the instrument logbook.

5.3.3. The software has pre-determined values that must be met in order to obtain a successful calibration. For additional details, refer to page 21 of the Orbis Vision User's manual (Rev 1.00, August 20, 2009).

5.4. Possible Sources of Error:

5.4.1. Low counts because sample is not in proper focus/orientation.

5.4.2. Background peak from rhodium window may interfere with weak chlorine peak.

5.4.3. Insufficient energy (kV setting) to knock electron out of the orbital.

6. References

EDAX Orbis Vision User's Manual, EDAX Inc., Mahwah, NJ, 2009.

Practical X-Ray Spectrometry, by Jenkins and deVries, distributed by Springer Verlag.

X-Ray Spectrochemical Analysis, by L.S. Birks, published by Interscience Publishers.

Visual Lines for Spectroscopic Analysis, by D.M. Smith, published by Hilger and Watts, Ltd.

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Worked Examples in X-Ray Analysis, by Jenkins and deVries, published by The Macmillan Press Limited.

Forensic Investigation of Explosions, Edited by Alexander Beveridge, published by Taylor & Francis Ltd.



ATF-LS-E5 X-ray Diffraction	ID: 775 Revision: 7
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1. Scope

- 1.1. X-ray diffraction (XRD), also known as X-ray powder diffraction, is used for the identification of any crystalline material, including but not limited to, inorganic and organic crystalline explosives, residues, raw materials, and fillers in adhesives and paint. Samples may be either individual substances or mixtures. X-ray diffraction is an identification technique for individual phases.

2. Instrumentation/Reagents

- 2.1. An X-ray diffractometer, as available in the laboratory. The instrument should be equipped with an ICDD database and appropriate software to allow automatic analyses including measuring, recording and comparison of patterns acquired with the desired databases. See parameter sheets for instrumentation and method specifics.
- 2.2. The calibration standards (Quartz, Silicon, and Corundum) are permanently prepared for analysis and should be stored appropriately.
- 2.3. Any user database patterns generated must be from known, properly documented sources or confirmed analytically by at least two other methods.
- 2.4. Patterns from the commercial ICDD database are from validated sources, mathematically calculated, or both.
- 2.5. New bottles of adhesion solvents should be tested by dropping several drops onto a zero-background plate, letting the solvent evaporate, and running the sample. New samples of other adhesion substrates should be applied to the sample holders in the manner normally used, and a sample run.

3. Safety Considerations

- 3.1. Safety precautions should always be used when grinding any substance, especially considering the potentially explosive nature of many of the samples normally encountered. For example, grinding small samples in a solvent such as hexane may lower the risk involved.
- 3.2. Safety glasses should be worn when working with powdered substances.
- 3.3. Sample preparation should be such that a minimum of sample can be scattered inside the instrument during the sample spinning.



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3.4. The inside of the instrument should be checked monthly and cleaned as needed to prevent sample build-up.

3.5. Also see radiation safety manual.

4. Procedure or Analysis

4.1. Sample Preparation

4.1.1. All samples should be run with as little sample preparation as possible to avoid rearrangement of anions and cations in a mixed sample. The specimen analyzed must be representative of the entire sample. Small particle size (allowing for a more random orientation of particles in the sample) is desired, so all samples should be well ground. Care should be taken when grinding potentially explosive materials.

4.1.2. Several different types of sample holders are available. An adhesion substrate such as a solvent, petroleum jelly, or double-sticky tape may be used to affix the sample to the holder when necessary. For some tape samples, the adhesive may be placed directly on the sample holder. A small piece of carbon tape can also be used to affix the tape sample to a low background slide, with the tape sample's adhesive face up.

4.1.3. In cases where crystal shape may result in "preferred orientation" of the crystals, additional steps may be taken to prepare the samples for analysis. Preferred orientation results in not all of the planes being detected in their predicted ratios (as in the case of long, flat crystals or fibers). More information can be found in the references, especially *Specimen Preparation For The Diffractometer* from Chapter 2 of the *ICDD X-ray Clinics manual* (6.3).

4.2. Procedures

4.2.1. After confirming that the calibration is current, samples are prepared by one of the methods addressed in Section 4.1. Using the XRD instrumentation and conditions given on the parameter sheets, a diffractogram is obtained. See instrument manuals or work instructions for further details on running samples. The raw data is then transferred into a format suitable for comparison to the commercial databases, and a search program is used to generate a list of possible matches. A manual search, using the Alphabetic, Fink, or Hanawalt Method, may also be used, either in addition to, or in place of, a computer search program.



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4.2.2. The diffraction patterns of the computer-suggested or search-found matches are then viewed by the examiner as a tool to help determine whether suggested components are actually present. Subtraction programs (either manual or computer based) may need to be used when multiple phases are present. A residue search can be performed on remaining peaks, which may aid in their identification. The diffractogram can also be compared to any standard diffractogram in the commercial or user-generated database to check for the presence of any individual phase. If possible, all lines should be accounted for. In the lab's experience, having only 1 or 2 matching lines is insufficient by itself to identify a compound. Therefore, at least 3 lines are required for pattern identification. For compounds having between 3 and 5 lines (such as aluminum), an attempt should be made to identify all lines in the pattern. Refer to "Using the Powder Diffraction File," published by ICDD, for guidelines on the criteria for a match.

4.3. Non-routine exams

4.3.1. Some samples may need to be handled in a non-routine manner. Solid objects, hard materials, and other non-routine samples can be handled using different sample holders. Specifically looking for a certain phase may require extending the region scanned, or may allow for a much quicker scan through a certain specified region. Any change from the stated operating parameters or from traditional sample preparation/sample holder, as well as a description of the need for the change, shall be documented in the case notes or on the instrument parameter sheet included in the case jacket.

5. Quality Assurance and Controls

5.1. When identifying a certain phase as being present, the diffractogram of that phase needs to be shown in the notes. This will assist in peer review and helps to assure quality results.

5.2. Variations in instrument performance will be demonstrated and adjusted, as necessary, during monthly calibration procedures.

5.3. Performance Checks

5.3.1. The performance check procedure will vary depending on the type of detector used with the instrument. A performance check shall be done within one month prior to analysis for casework, and following any instrument



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maintenance that could affect the performance. Procedures and acceptable conditions will be specified in work instructions for each instrument and the results will be recorded in the instrument logbook.

5.4. Validation

5.4.1. X-ray diffraction is a well-known, proven technique. The 1912 work of Max von Laue confirmed the wave character of X-rays by diffraction experiments from a single crystal. The field of X-ray crystallography followed from that work, with X-ray powder diffractometry being developed in 1935 by LeGalley and the first commercial equipment introduced by North American Philips in 1947. Changes since then have been primarily in the areas of detection and counting systems, automation, and the X-ray tubes themselves, with very few changes in the construction and geometry. Before an instrument is used for casework a validation/verification shall be performed to establish that its results are fit for purpose.

5.5. Limitations and Possible Sources of Error

- 5.5.1. Similar diffraction patterns can result from different compounds, and very complex patterns may result from mixtures. Amorphous (non-crystalline) substances, which would not give a diffraction pattern, could also be present. Compounds that comprise less than 1 % of a mixture may not be detected, but detection limits depend on the atomic number of the atoms in a compound, its crystal structure, and other properties of the sample.
- 5.5.2. The ICDD commercial databases utilize IUPAC nomenclature for the naming of chemical phases. Because this system is based on the atoms present, care should be taken to assure no confusion between different chemicals, which are given the same IUPAC name. A commonly found example is KClO_3 and KClO_4 , both named potassium chlorate in the databases. Different phases of the same chemical, such as anatase, rutile and brookite phases of TiO_2 , should also not be confused.
- 5.5.3. Peak displacement errors can occur if the sample is not level/flat or offset from the plane of the beam focus causing a consistent shift to the right. An example of this would be using too much sample in a low background slide. This displacement could hinder sample identification and may necessitate re-preparing and reanalyzing the sample. However it is possible that the software can compensate for this shift and re-preparing and reanalyzing can be avoided.



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- 5.5.4. As discussed in Section 4.1.3, preferred orientation of samples can result in changes in peak intensity ratios from what is expected. This should be taken into account when identifying phases or performing subtractions. These samples with multiple phases may need to be prepared again and rerun.

6. References

- 6.1. Introduction to X-Ray Powder Diffractometry, by Ron Jenkins and Robert L. Snyder, published by John Wiley and Sons, Inc.
- 6.2. Elements of X-Ray Diffraction, First and Second Editions, by B. D. Cullity, published by Addison-Wesley Publishing Company, Inc.
- 6.3. ICDD X-Ray Clinic Instruction Manuals, International Centre for Diffraction Data
- 6.4. X-Ray Diffraction Procedures, by Harold P. Klug and Leroy E. Alexander, published by John Wiley and Sons, Inc.
- 6.5. Reference Intensity Method of Quantitative X-Ray Diffraction Analysis, by Briant L. Davis, Institute of Atmospheric Sciences, South Dakota School of Mines and Technology
- 6.6. Practical X-Ray Spectrometry, by Jenkins and deVries, distributed by Springer-Verlag
- 6.7. X-Ray Spectrochemical Analysis, by L.S. Birks, published by Interscience Publishers
- 6.8. Visual Lines for Spectroscopic Analysis, by D.M. Smith, published by Hilger and Watts, Ltd.
- 6.9. An Introduction to X-ray Spectrometry, by Ron Jenkins, published by Heyden and Son, Ltd.
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7. Glossary

- Antiscatter slit - Controls the unwanted radiation that may pass through the receiving slit and strike the detector
- Beam mask - Retains a constant beam width, aiding the divergence slit in maintaining a constant exposure area
- Bragg's Law - Describes the relationship between the wavelength of the incident radiation, the angle of incidence, and the distance between the planes of the crystals
- Crystal lattice - Regular, repeating three-dimensional arrangement of atoms, resulting in predictable distances between the planes formed by the atoms
- Debye rings - Diffraction cones from a crystalline material that produce a set of concentric circles. Smooth and continuous rings indicate a fine-grained polycrystalline material. Continuous but spotty rings indicate a large-grained polycrystalline material. Individual spots and no rings indicate that the material is a single crystal.
- Detector slit - Aids beam monochromatization
- Diffractogram - Pattern resulting from scanning a sample over a specified range of angles of incidence (2θ values). Will include contributions from diffraction, scatter and fluorescence from the sample.
- Direct-beam center - X and Y coordinates (unwarped centroids) of the direct beam at a swing angle of zero
- Divergence slit - Adjusts aperture to maintain the same area of exposure on the sample during data collection
- GADDS - General Area Detector Diffraction System
- Göbel mirror - Graded multilayer mirror that creates a highly parallel incident beam while suppressing $K\beta$ radiation
- Monochromator - Gives improved peak/background ratios and suppression of $K\beta$ lines
- Phase - A unique arrangement of atoms in a regular, repeating arrangement. More than one phase may exist for the same chemical formula (e.g. rutile, brookite and anatase phases of TiO_2).



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- Phi rotation stage - Motorized circular stage that mounts to the XYZ stage and can rotate during sample collection. The rotation minimizes preferred orientation effects and is useful for small samples.
- Receiving slit - Controls the 2θ angle measured (resolution)
- Sample holder - Holds sample during analysis. Options include a low background slide, frosted glass slide, and cavity slide. The holder can vary depending on sample size and consistency. An automated version rotates at a predetermined constant rate to help compensate for surface effects and non-random crystal orientation.
- Sample-to-detector distance - Distance between the goniometer center and the flat front surface of the detector (where the fiducial plate mounts)
- Snout and pinhole slits - Similar to a collimator in that it focuses the X-ray beam. A snout and corresponding slit are attached to the Göbel mirror, which is attached to the X-ray tube.
- Soller slits - Contribute to reduced background and improved resolution by “straightening out” the X-ray beam
- X-ray diffraction - Utilizes a monochromatic beam of radiation to yield information about the d-spacings (distances between the planes) and their intensities from a single crystal or crystalline powder
- X-ray source - Tungsten filament emits electrons that bombard a target (in current operation – Cu anode is used). The target emits X-rays ($\text{Cu K}\alpha=0.154 \text{ nm}$) through beryllium windows towards the sample. Since the wavelength of $\text{Cu K}\alpha$ emission is similar to the interatomic distances in many crystalline materials, diffraction can occur.
- XYZ stage - Motorized, flat surface on which samples are placed. The stage is approximately 100 mm x 150 mm and can move in the X, Y, and Z (height) direction to align the sample. Motion of the stage in the XY direction during sample analysis can reduce the effects of preferred orientation.



ATF-LS-E6 Fourier Transform Infrared Spectroscopy	ID: 792 Revision: 8
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1. Scope

- 1.1. Fourier Transform infrared spectroscopy (FTIR) is a valuable method of identification and comparison in forensic exams. Both transmittance and reflectance techniques may be used for the analysis of a wide variety of organic and inorganic materials, such as explosives, plastics, adhesives, and trace evidence.
- 1.2. The absorption bands can provide enough characteristics to specifically identify a substance. The IR spectra of thousands of compounds have been collected, indexed and catalogued to serve as invaluable references for identifying substances.

2. Instrumentation/Reagents

- 2.1. The instruments available vary between laboratories. Any validated FTIR instrument can be used. See parameter sheets and/or spectral reports for details on instrumentation and settings.
- 2.2. Some FTIRs are equipped with two different detectors:
 - 2.2.1. The FTIR bench uses a DTGS (deuterated triglycine sulfate) detector which operates at room temperature.
 - 2.2.2. The FTIR microscope uses an MCT (mercury cadmium telluride) detector that must be cooled with liquid nitrogen.

3. Safety Considerations

- 3.1. Wear face shield or eye protection and protective gloves when handling liquid nitrogen
- 3.2. Use care when pressing a pellet (high pressures)

4. Procedure or Analysis

4.1. Sampling and Sample Prep

- 4.1.1. Sampling and sample preparation will vary depending on the physical state of the sample (e.g., solid, liquid, or gas) and the available accessories.
- 4.1.2. A background must be run before or after the sample, depending on the accessory. The accessory and conditions must be the same for the background and for the sample.



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5. Quality Assurance and Controls

5.1. Through regular calibration checks, use of known standards, and upkeep of instrument logbooks, the quality of the FTIR method is assured.

5.2. Instrument Performance

5.2.1. It is each analyst's responsibility to ensure that a polystyrene standard has been evaluated and an energy check has been performed within the last 31 days. Other tests of instrument performance may be run at the analyst's discretion.

5.2.2. There are four aspects of instrument performance that may be monitored:

1. Interferogram
2. Single-beam spectrum for microscope or bench
3. 100% T line
4. Polystyrene standard

Interferogram

- Refer to instrument manufacturer specifications. It may be useful to monitor interferogram position and voltage. Every detector is different so keeping track of the "normal" voltage is useful when trying to identify problems.
- It is a good practice to check the interferogram energy for each detector before use.

Single-Beam Spectrum

- The single beam spectrum of the open beam instrument (no accessories in the beam) can be diagnostic of instrument performance and alignment:
 - § Small peaks around 2900 cm⁻¹ indicate hydrocarbon contamination, probably oil contamination on the beamsplitter.
 - § Bands due to water (centered around 3750 and 1650 cm⁻¹) and carbon dioxide (around 2300 cm⁻¹) indicate the presence of these residual gases in the instrument. If these bands appear larger than normal after a reasonable purge time, the seals and purge setting should be checked.
 - § With an MCT detector, apparent absorption around 3300 cm⁻¹ may indicate the presence of ice in the detector (primary operator should be notified).

100% T Line



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- The 100 percent line is the ratio of two consecutive open-beam spectra and is used to measure system noise. Compare to the manufacturer's specifications for the noise in the monitored region. Increased noise may indicate fogged mirrors.

Polystyrene Standard

- This procedure confirms that selected peaks in a known spectrum correspond to the known wavenumber values of those selected peaks.
- Collect spectrum, in transmittance mode, for a known polystyrene standard.
- Compare the wavenumber values of the collected spectrum to the wavenumber values of known identifying peaks (as indicated on the standard card or in ASTM standard E1421-94). Values should not vary by more than 5-6 wavenumbers. Also, compare new wavenumber values to those of previous standard runs.

5.3. Possible Sources of Error

- 5.3.1. Excessive water and/or carbon dioxide interference due to wet sample or poor background.
- 5.3.2. Excessive noise in ATR spectrum due to inadequate contact between crystal and sample.
- 5.3.3. Complete absorbance (spectrum bottoms out) due to sample concentration (too concentrated) or pellet thickness (too thick).
- 5.3.4. Strongly absorbing crystals or mulls used in sample preparation may interfere with regions of the resultant sample spectrum.
- 5.3.5. Improper peak height ratios due to failure to perform ATR spectrum correction when necessary.

6. References

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- 6.2. Midkiff, C.R.; Washington, W.D., *Systematic Approach to the Detection of Explosives Residue, IV. Military Explosives*, *J. Ass. Off. Anal. Chem.*, Vol 59 (1976) p. 1357.
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- 6.4. *Yinon, J.; Zitrin, S., The Analysis of Explosives, Pergamon Series in Analytical Chemistry, Volume 3, Chapter 11.*
- 6.5. *Ryland, S. G., Infrared Microspectroscopy of Forensic Paint Evidence, Practical Guide to Infrared Microspectroscopy, Edited by Humecki H. J., 1995, p163-243.*
- 6.6. *Tungol, M.W.; Bartick E. G.; Montaser, A., Forensic Examination of Synthetic Textile Fibers by Microscopic Infrared Spectrometry, Practical Guide to Infrared Microspectroscopy, Edited by Humecki H. J., 1995, p245-285.*
- 6.7. *Colthup N. B.; Daly L. H.; Wiberley, S.E., Introduction to Infrared and Raman Spectroscopy, 3rd Edition, Academic Press, 1990.*
- 6.8. *Hopen T. J. Tricks of the Trade: Microtome Glass Knives Employed for Micro-FTIR Preparation, The Microscope, 50 (1), 2002, p4.*

7. Glossary

- Absorbance (A): The logarithm to the base 10 of the reciprocal of the transmittance (T).
 $A = \log_{10}(1/T) = -\log_{10}(T)$
- Absorption Spectrum: a plot or other representation of absorbance, or any function of absorbance, against wavelength or any function of wavelength.
- Attenuated Total Reflectance (ATR): reflection that occurs when an absorbing coupling mechanism acts in the process of total internal reflection to make the reflectance less than unity.
- Background: apparent absorption caused by anything other than the substance for which the analysis is being made.
- DTGS: Deuterated Triglycine Sulfate – refers to type of detector used on the FTIR bench.
- Far-Infrared: pertaining to the infrared region of the electromagnetic spectrum with wavelength range from approximately 400 to 10 cm^{-1} .
- Fourier transform (FT): a mathematical operation that converts a function of one independent variable to one of a different independent variable. In FT-IR spectroscopy, the Fourier transform converts a time function (the interferogram) to a frequency function (the infrared absorption spectrum). Spectral data are collected using an interferometer, which replaces the monochromator found in the dispersive infrared spectrometer.



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- Fourier Transform Infrared (FT-IR) spectrometry: a form of infrared spectrometry in which an interferogram is obtained; this interferogram is then subjected to a Fourier transform to obtain an amplitude-wavenumber (or wavelength) spectrum.
- Infrared: Pertaining to the region of the electromagnetic spectrum with wavelength range from approximately 0.78 to 1000 μm (wavenumber range 12,821 to 10 cm^{-1}).
- Infrared Spectroscopy: pertaining to spectroscopy in the infrared region of the electromagnetic spectrum.
- MCT: Mercury Cadmium Telluride – refers to the type of detector used on the microscope accessory.
- Mid-Infrared: pertaining to the infrared region of the electromagnetic spectrum with wavelength range from 4000 to 400 cm^{-1} .
- Spectrometer: Photometric device for the measurement of spectral transmittance, spectral reflectance, or relative spectral emittance.
- Transmittance (T): the ratio of radiant power transmitted by the sample, I, to the radiant power incident on the sample, I_0 .
- Wavelength: the distance, measured along the line of propagation, between two points that are in phase on adjacent waves.
- Wavenumber: the number of waves per unit length, in a vacuum, usually given in reciprocal centimeters, cm^{-1} .



ATF-LS-E9 Gas Chromatography-Mass Spectrometry (GC-MS)	ID: 795 Revision: 6
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1. Scope

1.1. This document provides guidelines establishing a common basis for the identification of analytes in explosive casework using gas chromatography-mass spectrometry (GC-MS). Specific instrumentation and procedures are provided in separate, individual work instructions kept near the GC-MS instrument they pertain to. In addition, detailed analytical conditions and instrumental settings for these methods shall be updated as necessary and kept on instrumental parameter sheets.

2. Instrumentation/Reagents

2.1. Any validated GC-MS instrumentation may be used for analysis. Since GC-MS systems come in a variety of configurations, the specific type of instrument, instrument settings, eluents, and required standard(s) are documented in individual work instructions and/or parameter sheets.

3. Safety Considerations

3.1. The following safety precautions will be considered when working with all GC-MS instruments:

Pump exhaust should be vented to a fume hood or other vent whenever facilities permit.

Used pump oil may contain chemicals that could pose potential health hazards and should be disposed of in an environmentally safe manner. Operators changing oil should wear laboratory coats, protective gloves and goggles or safety glasses.

All gases should be properly secured/stored. Pressure regulators should be inspected and examined for leaks whenever the cylinders are replaced.

Operators should exercise care when cleaning heated components.

At least two people should perform lifting of heavy equipment (vacuum pumps, etc.).

The hazards of chemicals and solvents must be understood before working with them.

The waste solvent containers should be emptied when full and solvents should be disposed of in an environmentally safe manner.

4. Procedure or Analysis

4.1. When filtering samples, appropriate filters (such as disposable syringe filters) that are compatible with the solvent shall be used. Detailed sample preparation guidelines,



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instrumental conditions, and procedures shall be located in the work instructions and/or parameter sheets for each method/instrument.

5. Quality Assurance and Controls

5.1. Tuning and Calibration

5.1.1. The mass spectrometer shall be tuned and/or calibrated according to manufacturer's recommendations. The frequency and procedure of tuning and/or calibration is instrument dependent and shall be outlined in the work instructions and/or parameter sheets for each GC-MS instrument. A primary operator or the manufacturer's engineers shall tune and calibrate the instrument after periodic maintenance and major instrument repairs. Documentation of all tuning, calibration, and periodic adjustments is required.

5.2. Parameter Sheets and Work Instructions

5.2.1. A copy of the parameter sheet detailing the instrument and analytical settings used in case work must be included in the case jacket. This will include at a minimum the date, mass analyzer, ionization source and polarity, reagents, eluents, column, injection volume, chromatographic and mass spectrometer settings/parameters. A list of the characteristic ions will be included on the parameter sheet or work instructions for that method.

5.3. Reference Standard

5.3.1. Each time a GC-MS instrument is used for analysis of a sample, a known reference material, such as an appropriate standard mixture containing the targeted analyte(s), shall be analyzed. The name and identifying information such as lot number and/or date prepared shall be documented.

5.3.2. The reference standard shall be analyzed at the beginning and end of a sequence of injections to assure the proper and continuous performance of the instrument during that sequence. A dated copy of one of the standards should be kept near the instrument. The reference standard data shall be evaluated to determine if the sensitivity/response, resolution, and peak shape are satisfactory, as defined in the work instructions.

5.4. Sample Analysis

5.4.1. Each time a GC-MS instrument is used for casework, an entry shall be



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documented in an instrument logbook. For any sample prepared for analysis related to casework an experimental material control shall be analyzed by the same method to determine potential contamination sources resulting from solvents, lab ware, concentration steps, or other sources. All unknown samples, material controls, and reference standard analyses shall be preceded by a solvent blank, which should be devoid of responses from the analyte(s) of interest. Any response to a target component in a solvent blank or control sample shall be documented and addressed in the case notes.

5.5. Instrument and Periodic Maintenance

- 5.5.1. All instrument maintenance that is necessary for proper instrument performance (column replacement, pump oil replacement, ion source cleaning, etc.) will be documented in an instrument maintenance log. The log will also document the symptoms and maintenance for any electrical component failures.

5.6. Requirements for Identification

- 5.6.1. At least four identification points are required to identify an analyte(s) of interest. One of these identification points shall be the retention time. Additional identification points can include characteristic ions and information from other detectors. The types of identification points that will be used are specific to a method and/or analyte, but the total number of identification points shall not be less than four. The following list outlines the requirements for each type of identification point:

5.6.1.1. Retention Time

Comparison of sample retention times to the reference standard retention times is required for the retention time to be used as a point of identification. The retention time of each targeted analyte will count as one point of identification if it agrees with that of the standard within 2% (or less, as determined by method validation). This is calculated as follows:

$$\frac{(\text{reference retention time} - \text{sample retention time})}{\text{sample retention time}} = -0.02 \text{ to } 0.02$$

The peak for the targeted component in the total ion chromatogram (TIC) or extracted ion chromatogram (EIC) shall have a signal-to-noise ratio of at least 3:1.



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5.6.1.2. Characteristic Ions

Characteristic ions can include the molecular ion or reproducible fragment ions and adducts. When possible, these ions should have a relative abundance exceeding approximately 10%, except for isotopes. For GC-MS/MS analysis, characteristic ions should have a relative abundance exceeding approximately 3%, except for isotopes. If possible, the molecular ion or pseudo-molecular ion should be selected as a characteristic ion.

Comparison of sample mass spectra (including relative abundance and signal-to-noise (S/N)) to the reference standard mass spectra is required for any characteristic ions used as points of identification. An extracted ion profile of a characteristic ion shall have a S/N threshold of at least 3:1. The mass spectrum from the unknown sample(s) should compare well to the spectrum obtained from the standard and there should be a general correspondence between the relative abundance for the unknown and standard (for example, within 10% of each ion's relative abundance). Any significant inconsistencies between the sample and the reference standard spectrum must be explained or an identification cannot be made. For example, any contamination from filters and solvent may be explained in the case jacket and not prevent an identification. Subtraction of background spectra is permissible and may be required to remove noise.

Each characteristic ion that meets these requirements will count as one point of identification. Therefore, with the retention time as one point of identification, only three characteristic ions would be needed for four total points of identification. If the mass spectrometer is capable of accurate mass measurements, two identification points may be assigned for each characteristic ion if the mass error is less than or equal to 5 ppm.

5.6.1.3. Information from Other Detectors

Additional identification points can also be attained through alternate ionization and/or chromatographic methods. For example, if an analyte is run on an EI method which gives 2 ions and then run on the same instrument/column with a CI method which gives 2 ions, this results in a total of 5 identification points. Another instrumental/analytical technique can also be utilized.

5.7. Sources of Error

- 5.7.1. A possible source of error can result from impurities in solvents and/or carryover from prior analyses, which can be eliminated through the analysis of standards, blanks, and material controls. The environmental conditions the instruments



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operate in can also cause gradual de-tuning/calibrating of the instrument. This can be eliminated by locating the instrument in a temperature and humidity-controlled room, using lock masses (if applicable), and/or using internal standards (if applicable). The sample concentration can also have an effect on the retention time and chromatographic separation, which can be addressed by diluting samples prior to analysis. An additional source of error could be co-eluting contaminants, which can obscure a response. This error is minimized, since the gas chromatograph is connected to a mass spectrometer.

6. References

Analysis of Organic Explosives, Propellants and Post-Blast Residues by Chemical Ionization GC/MS on an Ion Trap, P. A. Dreifuss and T. S. McConnell, *Proceedings of the 46th ASMS Conference on Mass Spectrometry and Allied Topics* (1998) 1475.

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Analysis of Explosives by Negative Ion chemical Ionization Mass Spectrometry, J. Yinon, *J. For. Sci.*, 25, No 2, (1980) 401-407.

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ATF-LS-E13 Ion Chromatography-Mass Spectrometry (IC-MS)	ID: 788 Revision: 6
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1. Scope

1.1. This document outlines general guidelines for utilizing Ion Chromatography-Mass Spectrometry (IC-MS) to identify inorganic monatomic and polyatomic ions and organic acids commonly found in explosives and their combustion products. Typically, this includes compounds with molecular weights less than 200 amu. IC-MS can be used to analyze aqueous extracts of both intact explosives and post blast evidence.

2. Instrumentation/Reagents

2.1. IC: Thermo Scientific Dionex ICS-5000⁺ (FSL-W) or ICS-6000 (FSL-A)

2.2. MS: Thermo Scientific ISQ EC

2.3. Software: Chromeleon 7

2.4. Since instrumentation and method specifics may vary by laboratory site, additional details concerning instrument modules, columns, analytical conditions, etc., can be found on IC-MS method parameter sheets. A parameter sheet will be present in each case jacket that includes IC-MS data.

3. Safety Considerations

3.1. Pump exhaust should be vented to a fume hood or other vent whenever facilities permit.

3.2. Used pump oil may contain chemicals that could pose potential health hazards and should be disposed of in a safe manner, e.g., Safety Kleen or other vendor. Operators changing oil should wear laboratory coats, protective gloves and protective eyewear.

3.3. Exercise care when cleaning or handling MS source components, which may be extremely hot.

3.4. Avoid reaching into the autosampler compartment if the autosampler arm or tray is moving.

4. Procedure or Analysis

4.1. Sample Analysis



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- 4.1.1. Every time the IC-MS is used, the operator's initials, date, and case number, at a minimum, will be recorded in the logbook.
- 4.1.2. Each sample set shall include a materials control, which is representative of all the procedures and materials used in preparing the sample.
- 4.1.3. Water blanks shall be run immediately before all materials controls and sample extracts and should be devoid of responses from the analyte(s) of interest. A response to a target component in a water blank or materials control shall be documented and addressed in the case notes or on the data. Due to the inherent sensitivity of the IC-MS, trace amounts of ions such as sulfate and chloride may be detected in water blanks or materials controls. The background levels of such ions must be taken into consideration when making identifications.
- 4.1.4. A standard shall be analyzed at the beginning and end of each sequence to assure the proper and continuous performance of the instrument and should be preceded by a water blank. The standard data shall be evaluated and documented as described in section 5.1.2.
- 4.1.5. Samples must be prepared using deionized water having a resistivity of 18.0 M Ω or higher, disposable plasticware (i.e., plastic beakers, pipets, syringes), and disposable syringe filters, preferably IC-specific filters.

4.2. Requirements for Identification

- 4.2.1. An IC-MS sample will be designated as positive for the presence of a specific inorganic ion or other targeted material if all the following conditions are met:

The retention time of the sample analyte agrees within 2-5% of that of the standard.

- Although retention times should fall within 2% of the standard when components are similar in concentration to that of the standard, concentration differences may cause sample retention to lengthen slightly if samples are very weak or shorten if samples are very concentrated.

The mass spectrum of the component is consistent with that of the standard.

Required characteristic ion(s) must be present; characteristic ions are defined either on the parameter sheet or in the work instructions.

Extracted ion chromatogram (EIC) must have a signal-to-noise ratio (S/N) of at least 3:1



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4.3. Non-routine exam – allowance for variation: changes to any of the IC-MS methods may be necessary for unique examinations of ionic species that may not be normally encountered yet need to be examined for a given case. Columns, eluents, and instrumental parameters may be changed to perform a unique analysis. However, any such changes must be fully documented and may require validation if they will be utilized in additional future casework.

5. Quality Assurance and Controls

5.1. Tuning and Calibration - The mass spectrometer shall be tuned and calibrated according to the manufacturer's recommendations. Tuning procedures can be found in the ISQ EC operator's and service manuals. The primary operator or the manufacturer's engineers should tune and calibrate the instrument after periodic maintenance, major instrument repairs, or at least once a year. Documentation of all tuning, calibration, and periodic adjustments is required.

5.1.1. The ISQ EC must pass tuning/calibration in both positive and negative mode.

5.1.2. Following tuning/calibration of the ISQ EC, a standard will be analyzed and the data evaluated. The IC-MS system can be used for the analysis of samples only after good quality mass spectra of the targeted species can be produced. Good quality mass spectra can be defined as spectra exhibiting the required characteristic ions in the absence of unexpected background interferences from masses not associated with that compound. This excludes the presence of known dimers, hydrates, etc., if they are typically observed for some anions. Chromatography and sensitivity (of both the mass spectrometer and conductivity detector) must also be acceptable as outlined in the work instructions. Verification of performance must be documented and may be accomplished by printing and initialing a copy of the standard to be kept in a logbook near the instrument, or by documenting the review and any issues in the user logbook, for example.

5.2. Reference Materials

5.2.1. Reference materials for IC-MS include the MS calibrant solution, as well as ionic stock solutions and working standards (either purchased or prepared in-house).



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In-house working standards are made from standard ppm stock solutions prepared from certified chemicals obtained from a reputable chemical supplier, such as Fisher Scientific, VWR, Sigma-Aldrich, etc.

Documentation for in-house standards and stock solutions must include:

- Date prepared and by whom.
- Compound name, manufacturer, and lot number of chemicals used.
- How standards and stock solutions were prepared (weight, volume, concentration, etc.).

Documentation for purchased standards or calibrant solutions must include:

- Date opened and by whom.
- Compound name, manufacturer, and lot number.

5.3. Instrument and Periodic Maintenance - All instrument maintenance necessary for proper IC-MS performance (e.g., column replacement, pump oil replacement, ion source or analyzer cleaning, etc.) will be documented in an IC-MS maintenance log. The log will also document the maintenance and symptoms from any electrical component failures that might occur.

5.4. Primary Operators should perform and record maintenance (or request service) to correct any of the following:

- Loss of resolution
- Loss of retention
- Loss of detector response
- Abnormal peak shapes
- Abnormal operating pressures

5.5. Possible sources of error

5.5.1. Possible sources of error include impurities from solvents and/or carryover from prior analyses. Other possible sources of error include calibration errors and gradual de-tuning of the MS, which normally do not contribute to the generation of false positives. These sources can be eliminated through the analysis of standards, blanks and materials controls. Also, temperature and sample concentration may affect retention time and chromatographic separation. This is addressed by keeping the column compartment at a constant temperature, and by diluting samples prior to analysis.



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- 5.5.2. Environmental - The sensitivity of the IC-MS and the ability to confirm trace levels of materials necessitate a level of awareness of anions that are commonly found in the environment at trace levels.

6. References

Thermo Scientific hardware manuals for ISQ EC, ICS-5000 or ICS-6000 and Chromeleon software.

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ATF-LS-E15 Ignition Susceptibility Test	ID: 777 Revision: 5
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1. Scope

1.1. Ignition is the act of kindling or setting on fire any combustible substance. An ignition susceptibility test (IST) is conducted to determine if suspected intact explosives or fuse will support combustion. Many of these materials exhibit characteristic burning behavior when ignited. Although additional testing is required for positive identification, the burning characteristics may be useful indicators of the identity of the suspect material.

2. Instrumentation/Reagents

2.1. Tweezers, spatula, or other suitable instrument

2.2. Ignition source (burner, match, etc.)

2.3. A reference material of known explosive may be used for comparative purposes. If a reference material is used, the lot number or unique identifier must be noted.

3. Safety Considerations

3.1. Use as small a sample as possible. Make sure the area is clear of any flammable or explosive materials (solvents, containers of explosives, etc.). Perform test in a hood, if practical, and wear eye protection.

4. Procedure or Analysis

4.1. Remove a small representative sample of the suspected explosive. Using a spatula or other appropriate tool, introduce the sample to an ignition source (flame) and observe the resulting effect. Make note of the physical changes that occur in the sample, such as flame/flash colors, smoke, sound (gas release), remaining residues, etc.

4.2. A length of fuse can be affixed to the end of a probe or held in tweezers and ignited. The observed burning characteristics that demonstrate the functionality of the fuse shall be noted.

4.3. Some compounds and energetic mixtures will not burn unconfined in an open flame. Light confinement in a rolled piece of tissue paper may be helpful in the ignition of these mixtures. It should be noted that a negative IST result does not necessarily indicate the reactivity of a substance. Some explosives will not ignite even if confined.



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4.4. Extreme caution must be exercised when testing suspected primary high explosives as a very small sample will release a great amount of energy.

5. Quality Assurance and Controls

5.1. The physical condition of the sample, along with the ignition technique, need to be taken into consideration if the sample does not burn as expected. Conditions that may interfere include excessive moisture, mixtures or impure samples, and improper ignition technique.

6. References

6.1. *Fedoroff, B.T.; Sheffield, O.E. Encyclopedia of Explosives and Related Items, PATR 2700, Picatinny Arsenal, Dover, NJ, Vol. 7, 1975, p 111.*

6.2. *Washington, W.D.; Midkiff, C.R., Systematic Approach to the Detection of Explosive Residues. I. Basic Techniques, Journal of the AOAC, 55 (4), 1972, p. 811-822.*

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ATF-LS-E17 Spot Tests	ID: 785 Revision: 5
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1. Scope

- 1.1. Spot tests are used for testing/screening of suspected explosive ingredients, residues, or precursor materials, such as acids. Colors and/or crystalline precipitates resulting from reactions of a known reagent with an unknown solution are used for identification of anions or cations. See attached procedures for each specific spot test.

2. Instrumentation/Reagents

- 2.1. Containers such as spot plates, glass well slides, vials, test tubes, etc.
- 2.2. Disposable pipettes
- 2.3. UV light and hot plate (if needed)
- 2.4. Standard solutions and/or reagents- see attached procedures

3. Safety Considerations

- 3.1. Follow chemical hygiene plan.
- 3.2. Wear eye protection and gloves.

4. Procedure or Analysis

- 4.1. Dropwise addition of test reagent to sample (extract, solution, residue, etc.) in a spot plate, well slide, vial, or other appropriate container/vessel. Examine formation of characteristic color or precipitate and record actual observations as well as conclusions in the case notes. See attached procedures for the specific spot test to be completed.
- 4.2. Non-Routine Exam
 - 4.2.1. Minor modifications, (e.g., adjustments in the number of drops applied depending on sample volumes) are acceptable and may be necessary based on the circumstances of the case. Any significant modifications in procedure must be properly documented.

- 4.3. See the following procedures for the specific spot test to be completed:

<u>Aluminum Spot Test - Morin Reagent</u>	3
<u>Ammonium Ion in Water Extracts - Nessler Spot Test</u>	4
<u>Chloride in Water Extracts - Silver Nitrate Spot Test</u>	5
<u>Hypochlorite - Potassium Iodide Color Test</u>	6
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5. Quality Assurance and Controls

- 5.1. Use disposable supplies whenever possible to avoid contamination.
- 5.2. When using spot plates and well slides, avoid overfilling the well.
- 5.3. Use reagents that have not expired and that are properly labeled with date, contents, and examiner initials.
- 5.4. Run positive and negative controls using known references or standards and the same materials used for sample analysis (same source of well slides, vials, pipettes, etc.)

6. References

See attached procedures for relevant references.



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Aluminum Spot Test - Morin Reagent

1. Principle

- 1.1. Aluminum salts and aluminum in neutral or slightly acidic solution reacts with Morin reagent to form a complex that produces intense green fluorescence under UV light. Interferences include free and salts of beryllium, indium, gallium, thorium, scandium, and zirconium.

2. Reagents

- 2.1. Morin reagent (3,5,7,2',4'-pentahydroxyflavanone), saturated solution in alcohol
- 2.2. 10 % NaOH (aqueous)
- 2.3. Dilute acid

3. Determination

- 3.1. Add 2 drops 10 % NaOH to a spot plate, well slide, or other appropriate vessel for viewing color change.
- 3.2. Add small amount of suspected aluminum. The alkaline solution digests the aluminum with the evolution of hydrogen gas. This digestion may be noted macroscopically and/or microscopically.
- 3.3. Add diluted acid to aqueous test solution to make the solution slightly acidic.
- 3.4. Add 2 drops of Morin reagent and observe under UV light. A fluorescent green color is a positive test result.

4. Detection Limit

- 4.1. 0.2 µg/mL aluminum (5.1)

5. References

- 5.1. Houghton, R., Field Confirmation Testing for Suspicious Substances, CRC Press, Taylor & Francis Group, 2009, p. 277-278.
- 5.2. Jungreis, E., Spot Test Analysis: Clinical, Environmental, Forensic, and Geochemical Applications, 2nd Edition, Wiley-Interscience, 1997, p. 89.
- 5.3. Paul, A.; Gibson, J., Qualitative Analysis for Aluminum, Journal of Chemical Education, 1962, 39(8), p. 398.



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Ammonium Ion in Water Extracts - Nessler Spot Test

1. Principle

- 1.1. Most tests for the ammonium ion are based on detection of NH_3 liberated in strongly basic media. Ammonia reacts with a mixture of KI and HgI_2 to form an orange-red precipitate of $\text{HgI}_2 \cdot \text{HgNH}_2\text{I}$. As a spot test, the reaction directly in alkaline solution produces an orange-brown precipitate indicative of ammonium salts. The reagent may be prepared in the laboratory (5.2) or purchased pre-mixed. Sulfate and sulfide are reported to interfere but may be avoided by conducting the test in two steps (5.1).

2. Reagents

- 2.1. Nessler's reagent – CAS 7783-33-7

3. Determination

- 3.1. Pipet 1-2 drops of a neutral or slightly acidic water extract solution to a white spot plate, a well slide, or other appropriate vessel that allows viewing a dark-colored precipitate.
- 3.2. Add 1-2 drops of Nessler's reagent.
- 3.3. An orange-brown precipitate is indicative of an ammonium salt.

4. Detection Limit:

- 4.1. Reported: 0.3 ppm NH_3 (5.1)

5. References

- 5.1. Feigl, F.; Anger, V., Spot Tests in Inorganic Analysis, Elsevier Publishing Co., New York, N.Y., 1972, p. 337.
- 5.2. Benedetti-Pichler, A.A., Identification of Materials, Academic Press, New York, N.Y., 1964, p. 366.



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Chloride in Water Extracts - Silver Nitrate Spot Test

1. Principle

1.1. Silver nitrate produces turbidity in solution at ppm levels and forms white precipitates with higher levels of chloride and thiocyanate in acidic medium. SCN^- is readily identified with a ferric chloride test in a separate sample or using acetic acid and peroxide to prevent coprecipitation of SCN^- (4.1). SCN^- can also be removed by oxidation. Other halo anions such as bromide and iodide also precipitate but are colored. Feigl describes use of hydrogen peroxide and 8-hydroxyquinoline (HQ) to eliminate interference by Br^- and I^- (4.1). Sulfide, if present, interferes but can be readily removed.

2. Reagents

- 2.1. Silver nitrate solution - 1 % aqueous. Dissolve 1 g ACS reagent grade AgNO_3 in 100 mL of ultra pure water (UPW). Store in brown bottle to ensure stability.
- 2.2. Nitric acid - 6N. Dilute 20 mL ACS reagent grade HNO_3 to 50 mL with UPW.

3. Determination

- 3.1. Pipet 4-5 drops of water extract solution to a vial, test tube or other appropriate container.
- 3.2. Acidify with 1-2 drops 6 N HNO_3 .
- 3.3. Add dropwise 2-3 drops 1 % AgNO_3 solution, shake and observe turbidity or precipitate.

Very faint turbidity usually lacks analytical significance.

If significant precipitate is observed, test for SCN^- .

If a brown/black precipitate forms, on a second aliquot of the extract, acidify with 4-5 drops 6N HNO_3 , heat to boiling for a few seconds, cool and retest with AgNO_3 . The dark precipitate should not be observed, and a white precipitate will be formed if chloride is present.

4. References

- 4.1. Feigl, F. and Anger, V. Spot Tests in Inorganic Analysis, Elsevier Publishing Co., New York, N.Y., 1972, p. 638, 178-179.
- 4.2. Benedetti-Pichler, A.A. Identification of Materials, Academic Press, New York, N.Y., 1964, p. 389.
- 4.3. Riss, D., [Testing for Chlorides with Silver Nitrate](#) (PDF format), Conserve O Gram 6/3, Washington, D.C., National Park Service, 1993.



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Hypochlorite - Potassium Iodide Color Test

1. Principle

- 1.1. Hypochlorite can oxidize heavier halides to free halogens in acidic medium. Advantage can be taken of the high solubility of free halogens in halogenated solvents such as CCl_4 relative to water, to concentrate liberated bromide or iodide and enhance visualization of a characteristic color. Levels of either bromide or iodide relative to one another could interfere by color masking.

2. Reagents

- 2.1. Potassium iodide solution - 0.5 M. Dissolve 8.30 g ACS reagent grade KI in 100 mL UPW.
- 2.2. Hydrochloric acid - 6N. Dilute 25 mL ACS reagent grade HCl to 50 mL with UPW.

3. Determination

- 3.1. Dissolve a small amount of the suspected solid in UPW or pipet 8-10 drops of a suspected hypochlorite solution into a vial, test tube or other appropriate container.
- 3.2. Acidify with 2-3 drops of 6 N HCl.
- 3.3. Add dropwise 4-5 drops of 0.5 M KI solution followed by 6-8 drops of CCl_4 .
- 3.4. Shake for a few seconds and allow layers to separate.
- 3.5. Observe presence of color in the CCl_4 layer. A pale to dark lavender color, depending on concentration, indicates hypochlorite.

4. Detection Limit

- 4.1. Not determined



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Nitrate in Water Extracts – Modified Griess Test

1. Principle

- 1.1. Nitrites react with primary amines in mildly acidic solutions to form diazonium cations, but nitrates do not. Once formed, the diazonium cations react further to produce colored azo dyes. With an active metal such as zinc, in an acid medium, nitrates can be reduced to nitrites which then react to form the azo dyes. Interferences, if any, are rare. Because of the color intensity, detecting small amounts of nitrate in the presence of nitrites is difficult. Some indication of the presence of nitrates may be seen by diluting the sample and testing two equivalent portions side-by-side. Zinc is added to one and not the other. If the zinc-containing sample has more intense color, it may contain nitrates in addition to nitrites.

2. Reagents

- 2.1. Sulfanilic acid solution - dissolve 1 g ACS reagent grade sulfanilic acid by warming in 100 mL 30 % acetic acid (aqueous).
- 2.2. N-1-naphthylethylenediamine dihydrochloride (NED·2HCl) solution - dissolve 1 g ACS reagent grade NED·2HCl in 100 mL 70/30 ethanol/acetic acid solution. Store in a brown glass bottle for stability.
- 2.3. Zinc powder, fine

3. Determination

- 3.1. Pipet 4-5 drops of water extract solution to a vial, test tube, etc., or 2-3 drops to a white spot plate, well slide, or other appropriate vessel that allows viewing a color change.
- 3.2. Add 1-2 drops of sulfanilic acid solution followed by 1-2 drops of naphthylethylenediamine solution. If little color develops, add a small amount (a few mg) of zinc powder.
A rose to deep red color developing in the vicinity of the zinc powder indicates nitrates.
With high levels, color develops rapidly but low levels may require a minute or more for color development to become apparent.

4. Detection Limit

- 4.1. 0.05 ppm, as nitric acid (5.1)

5. References

- 5.1. Feigl, F. and Anger, V., Spot Tests in Inorganic Analysis, Elsevier Publishing Co., New York, N.Y., 1972, p. 363.
- 5.2. Jungreis, E., Spot Test Analysis Clinical, Forensic and Geochemical Applications, John Wiley & Sons, New York, N.Y., 1985, p. 207-209.



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Sulfate in Water Extracts – Barium Chloride Spot Test

1. Principle

- 1.1. Barium chloride produces turbidity in solution at ppm levels and forms a white precipitate with higher levels of sulfate in acidic medium. Common barium salts of other anions are soluble in acid and do not interfere. This test is specific (4.1).

2. Reagents

- 2.1. Barium chloride solution – 5 % aqueous. Dissolve 5 g ACS reagent grade BaCl_2 in 100 mL UPW.
- 2.2. Hydrochloric acid - 6N. Dilute 25 mL ACS reagent grade HCl to 50 mL with UPW.

3. Determination

- 3.1. Pipet 4-5 drops of water extract solution to a vial, test tube or other appropriate container.
- 3.2. Acidify with 1-2 drops 6 N HCl.
- 3.3. Add dropwise 3-4 drops 5 % BaCl_2 solution, shake and observe turbidity or precipitate. If no initial turbidity or precipitate is observed, set sample aside and recheck in 10-15 minutes. With relatively low levels of sulfate, precipitate may form slowly. Heating of the sample may also enhance precipitate formation.

4. References

- 4.1. Benedetti-Pichler, A.A., Identification of Materials, Academic Press, New York, N.Y., 1964, p. 381.



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Sulfide in Water - Silver Nitrate Spot Test

1. Principle

- 1.1. Silver nitrate gives a brown/black precipitate with low levels of sulfide (or hydrosulfide) in dilute acidic medium. Aqueous sulfide(s) are readily oxidized to sulfate by heating with dilute HNO_3 . In acidic medium, silver sulfate is soluble, so no precipitate is observed.

2. Reagents

- 2.1. Silver nitrate solution - 1% aqueous. Dissolve 1 g ACS reagent grade AgNO_3 in 100 mL UPW. Store in brown bottle to ensure stability.
- 2.2. Nitric Acid - 6N. Dilute 20 mL ACS reagent grade HNO_3 to 50 mL with UPW.

3. Determination

- 3.1. Pipet 4-5 drops water extract solution to a vial, test tube or other appropriate container.
- 3.2. Acidify with 1-2 drops 6 N HNO_3 .
- 3.3. Add dropwise 2-3 drops 1 % AgNO_3 solution, shake and observe turbidity or precipitate.
- 3.4. If turbidity is observed which darkens in a few seconds, continue observation.
- 3.5. If the precipitate becomes brown/black, test a fresh sample of the original extract, acidify with 4-5 drops 6N HNO_3 , heat to boiling for a few seconds, cool and retest with AgNO_3 .
A white precipitate of AgCl indicates that both sulfide(s) and chloride are present in the sample.
No precipitate indicates sulfide(s) only.

4. Detection Limit

- 4.1. Not determined



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Sulfur Spot Test

1. Principle

- 1.1. Crystalline free sulfur is extracted from the questioned material with pyridine. The color observed will vary from blue or green for low concentrations to brown for high concentrations. Interferences include water and aluminum. Hydrocarbons and CS₂ inhibit color formation. Polyhalogenated hydrocarbons, CHCl₃, Cl₂, Br₂, and I₂ also interfere.

2. Reagents

- 2.1. Pyridine
- 2.2. 10 % NaOH – dissolve 10 g NaOH in 100 mL UPW.

3. Determination

- 3.1. Place sample of material suspected to contain sulfur in a vial, test tube or other appropriate container.
- 3.2. Add several drops of pyridine and gently warm.
- 3.3. Remove from heat and add approximately one-tenth its volume of sodium hydroxide solution. The formation of a blue, green, or brown color indicates the presence of sulfur. The color will begin to fade after a few moments.
- 3.4. If no particles of sulfur are visible or if the sample contains powdered metal, wash the dry solid with pyridine, collect the filtrate and test with sodium hydroxide as above. A disposable glass pipet with glass wool plugs can be used as a filtering mechanism.

4. Detection Limit

- 4.1. Not determined. According to Lange (5.1), sulfur is detectible at a concentration of 1:300,000 in pyridine.

5. References

- 5.1. Lange, E., Handbook of Chemistry, 9th Edition, Handbook Publishers, Inc., 1956, p. 981-982.
- 5.2. Meyers, R., A Systematic Approach to the Forensic Examination of Flash Powders, Journal of Forensic Sciences, 1978, 23(1).
- 5.3. Jungreis, E., Spot Test Analysis: Clinical, Forensic and Geochemical Applications, John Wiley & Sons, New York, N.Y., 1985 p. 69.



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Thiocyanate in Water - Ferric Chloride Spot Test

1. Principle

- 1.1. Thiocyanate and chloride both give white precipitates with AgNO_3 in acidic medium. Ferric Chloride solution gives a rose to blood red color with thiocyanate in slightly acidic or neutral aqueous solutions. Color hue and intensity are dependent on SCN^- concentration.

2. Reagents

- 2.1. Ferric chloride solution - 10 % aqueous. Dissolve 1 g ACS reagent grade FeCl_3 in 10 mL UPW. Store in brown bottle to ensure stability.

3. Determination

- 3.1. Pipet 1-2 drops water extract solution to a white spot plate, a well slide, or other appropriate vessel that allows viewing a color change.
- 3.2. Add dropwise 2-3 drops 10 % FeCl_3 solution and observe color development.
 - A faint rose color indicates low levels of SCN^- and an intense red, much higher levels. If the original extract solution is highly colored, traces of SCN^- may not be observed.
 - Testing of lightly colored extracts can be facilitated by using, in an adjacent spot plate well, an equivalent amount of extract solution, and adding water equivalent to the volume of the amount of FeCl_3 solution used.
 - § Colors of the control and test samples are compared.
 - § If desired, an additional sample is acidified with 4-5 drops 6N HNO_3 and heated to boiling for a few seconds. The cooled sample is then retested with FeCl_3 , a clear yellow, rather than red, color is indicative of SCN^- .
 - § Similar results are obtained with oxidation of the SCN^- and testing with silver nitrate, i.e., no white precipitate.

4. References

- 4.1. Curtman, L.J., Semimicro Qualitative Chemical Analysis, Macmillan Co., New York, N.Y., 1954, p. 297.
- 4.2. Benedetti-Pichler, A.A., Identification of Materials, Academic Press, New York, N.Y., 1964, p. 361.



ATF-LS- E19 Stereomicroscopes	ID: 780 Revision: 6
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1. Scope

- 1.1. This document describes the procedure for basic maintenance of stereomicroscopes and calculation of ocular scale magnification factors.

2. Instrumentation/Reagents

- 2.1. A wide field binocular stereomicroscope capable of manipulating small samples encountered as evidence
- 2.2. A source of illumination (reflected incident light source)
- 2.3. An ocular/eyepiece micrometer
- 2.4. A stage micrometer with known graduated units

3. Procedure or Analysis

3.1. Calculating the ocular scale magnification factor:

- § Insert the eyepiece micrometer into the ocular. If the ocular piece can be focused, focus the ocular in such a manner that the scale appears with maximum sharpness.
- § Place the stage micrometer under the stereomicroscope, illuminate and focus the stereomicroscope on the scale. Both ocular and stage micrometers should be sharply defined.
- § Turn the eyepiece until both scales are parallel to each other.
- § Determine how many divisions of the ocular micrometer correspond to a certain distance on the stage micrometer and calculate the length which corresponds to one division of the ocular micrometer. Note: If all the points on the stage micrometer are not certified/calibrated, then only the certified/calibrated points shall be used.
 - § Example: 70 divisions on the ocular micrometer corresponds to 0.4 mm (or 400 um) on the stage micrometer.

$$\text{One division} = \frac{400 \text{ um}}{70 \text{ divisions}} = 5.7 \text{ um}$$



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§ Thus if an item measures 20 divisions wide on the ocular scale, the magnification factor of 5.7 um can be used to calculate the item's width of 114 um.

§ The calculated ocular scale magnification factor applies only to the specific ocular, objective and stereomicroscope dial setting (such as 40x) for which the measurement was made.

§ The above procedure should be repeated for each dial setting available on the individual stereomicroscope. The calculated ocular scale magnification factors can be taped to the stereomicroscope for reference.

3.2. Refer to the document on maintenance and performance monitoring of measuring devices (ATF-LS-E21) to determine how frequently to perform this procedure.

4. Quality Assurance and Controls

4.1. If the precision tracks in the stereomicroscope become dirty, maintenance should be performed by a manufacturer's representative under service contract.

4.2. Dust on optical elements will degrade the image quality to some extent. The best way to avoid dust exposure is to keep the stereomicroscope covered. The stereomicroscope tube or housing should always be closed with objectives and oculars in place.

4.3. To maintain sharpness of image and contrast, objectives and oculars should be cleaned, and checked for cracks as necessary.

4.4. Dust, fingerprints, or other oils can be removed with a dust free rag and breathing on the optical surface. If a solvent is necessary, use as little alcohol as possible to remove oils. Compressed air can be used to blow dust from optical parts as well.

5. References

5.1. Mollring, F.K., Microscopy from the Very Beginning, Carl Zeiss Microscopy Laboratory, Oberkockbien, Germany.

5.2. McCrone, W.C., et al., Polarized Light Microscopy, Ann Arbor Science Publishers Inc, Ann Arbor, MI, 1978.



ATF-LS-E21 Maintenance and Performance Monitoring of Measuring Devices	ID: 781 Revision: 7
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1. Scope

- 1.1. This document defines the policies, work instructions, schedule and documentation for the maintenance and performance monitoring of measuring devices and any external calibrations required in the trace evidence discipline.
- 1.2. This document is applicable to the trace evidence discipline of the ATF Forensic Science Laboratories, which includes the Explosives Section, the Fire Debris Section and the Trace Evidence Section.
- 1.3. Unless otherwise stated in a discipline-specific method or this procedure, the measurements documented in the trace evidence discipline are recorded for comparative or descriptive purposes only. It has been established that these measurements do not significantly affect the test result and that the associated contribution from a calibration would contribute little to the total uncertainty of the test result. Accordingly, measuring devices used for these measurements do not require calibration. These measurements are regarded as approximate or nominal and when applicable, should be reported as such.
- 1.4. The ATF laboratory ensures that the equipment used for all measurements can provide the uncertainty of measurement needed by adhering to the guidelines in this protocol.

2. Procedure or Analysis

- 2.1. Measuring devices with movable parts, such as calipers and micrometers, shall be performance checked and/or verified according to the established schedule. When using one of these devices, the examiner should be able to identify which measurement device was used, either by recording the unique identification number in the case notes or by always using an assigned tool.
- 2.2. ATF Forensic Science Laboratories have determined that when reporting approximate or nominal measurements in the trace evidence disciplines, rulers and other instruments that do not have movable parts do not have a significant effect on the test result; therefore they are exempt from this requirement. Rulers and other instruments without movable parts must be replaced when they exhibit notable wear or damage.
- 2.3. Work Instructions:

Calipers and Micrometers

- Use at least three different blocks that represent the range of measurements typically taken with the tool. (Pick blocks that are at least 0.010 inch apart for micrometers and 0.25 inch apart for calipers).



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- Insert the center of the block into the caliper/micrometer and measure.
- Record the size of the gauge block used and the measurement obtained.
- If the caliper/micrometer is within the tolerance on all measurements, the tool has been verified. If the caliper does not measure within the tolerance, the caliper should be re-set and repeat steps one through three. The tolerance on micrometers is 0.003 inch and on calipers 0.05 inch.

Balances (in-house performance checks)

- Use a certified weight set.
- Use at least three different weights on the balance that span the range of weights.
- The balance is considered to be within tolerance when the weights are within ± 10 times the readability of the balance.
- Record the weights used and measurement obtained.

3. Quality Assurance and Controls

3.1. Performance Check / Calibration schedules:

Microscopes – The microscope’s ocular scale is often used in a comparative manner, such as in comparing the number of ocular scale divisions in the width of two fibers. If a measurement is to be described in units other than ocular scale divisions, such as millimeters, then a calibrated stage micrometer is used to calculate the ocular scale magnification factor. Once established, further performance checks of a microscope are not required unless the microscope has been moved or damaged. If the stage micrometer becomes damaged, it shall be taken out of service and sent for a quality check by an external calibration lab. If all of the points on the stage micrometer are not certified/calibrated, then only the certified/calibrated points shall be used. Work instructions for calculating the ocular scale magnification factor are in *ATF-LS-E19 Stereomicroscopes* and *ATF-LS-TE02 Setup and Use of the Microscope*.

Balances – Balances will either be calibrated annually by an external vendor or have an in-house performance check at least once a year using a certified weight set. Work instructions for the in-house performance check are noted in section 2.3. The weight set used for in-house performance checks will be sent to an external calibration lab on a regular schedule. The scheduled interval shall not exceed 10 years.

Calipers and micrometers – Calipers and micrometers will be subjected to a performance check at least once a year with a calibrated gauge block set. Work instructions for those performance checks are noted in section 2.3. The gauge



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block sets used for performance checks will be sent to an external calibration lab on a regular schedule. The scheduled interval shall not exceed 10 years.

3.2. Logbooks will be reviewed during the annual internal quality reviews.

4. References

- 4.1. ATF-LS-6.5 Metrological Traceability
- 4.2. ATF-LS-6.6 Externally provided products and services
- 4.3. Instrument/equipment-specific manufacturer manuals



ATF-LS-E23 Liquid Chromatography-Mass Spectrometry (LC-MS)	ID: 3358 Revision: 5
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1. Scope

1.1. This document outlines general guidelines for utilizing liquid chromatography-mass spectrometry (LC-MS) technologies (including HPLC, UPLC/UHPLC, MS, and MS/MS) to identify compounds in explosive casework. Specific instrumentation and procedures are provided in separate, individual work instructions kept near the LC-MS instrument they pertain to. In addition, detailed analytical conditions and instrumental settings for these methods shall be updated as necessary and kept on instrumental parameter sheets.

2. Instrumentation/Reagents

2.1. Any validated LC-MS instrumentation may be used for analysis. Since LC-MS systems come in a variety of configurations, the specific type of instrument, instrument settings, eluents, and required standard(s) are documented in individual work instructions and/or parameter sheets.

3. Safety Considerations

3.1. The following safety precautions will be considered when working with all LC-MS instruments:

Pump exhaust should be vented to a fume hood or other vent whenever facilities permit.

Used pump oil may contain chemicals that could pose potential health hazards and should be disposed of in an environmentally safe manner.

All gas cylinders should be properly secured and stored. Pressure regulators should be inspected and examined for leaks whenever the cylinders are replaced.

Avoid reaching into an autosampler compartment when the injector needle is moving.

Operators should exercise care when cleaning heated components.

At least two people should perform lifting of heavy equipment (vacuum pumps, etc.)

The waste solvent containers should be emptied when full and solvents should be disposed of in an environmentally safe manner.

4. Procedure or Analysis

4.1. For all LC-MS methods, sample extracts should be diluted with running solvents/buffers when necessary. If diluted with other solvents/buffers, the retention times of the analytes of interest could be significantly altered from previous standard data and additional ions could be present in the mass spectral data. Filtration of samples with disposable syringe



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filters is also recommended, especially if a UPLC/UHPLC instrument is utilized. Detailed sample preparation guidelines, instrumental conditions, and procedures shall be located in the work instructions and/or parameter sheets for each method/instrument.

5. Quality Assurance and Controls

5.1. Tuning and Calibration

5.1.1. The mass spectrometer shall be tuned and calibrated according to manufacturer's recommendations. The frequency and procedure of tuning and calibration for each LC-MS instrument shall be outlined in the work instructions and/or parameter sheets if completed by the primary operator and/or users. A primary operator or the manufacturer's engineers should tune and calibrate the instrument after periodic maintenance, major instrument repairs, or at least once a year. Documentation of all tuning, calibration, and periodic adjustments is required.

5.2. Parameter Sheets and Work Instructions

5.2.1. A copy of the parameter sheet detailing the instrument and analytical settings used in case work must be included in the case jacket. This will include at a minimum the date, mass analyzer, ionization source and polarity, reagents, eluents, column, injection volume, chromatographic and mass spectrometer settings/parameters. A list of the characteristic ions will be included on the parameter sheet or work instructions for the method. Minor adjustments to instrument settings are common in LC-MS and the parameter sheet shall be updated when applicable.

5.3. Reference Standard

5.3.1. Each time a LC-MS instrument is used for analysis of a sample, a known reference material, such as an appropriate standard mixture containing the targeted analyte(s), shall be analyzed. The name and identifying information such as lot number and/or date prepared shall be documented.

5.3.2. The reference standard shall be analyzed at the beginning and end of a sequence of injections to assure the proper and continuous performance of the instrument during that sequence. A dated copy of one of the standards should be kept near the instrument. The reference standard data shall be evaluated to determine if the sensitivity/response, resolution, and peak shape are satisfactory, as defined in the work instructions.



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5.4. Sample Analysis

5.4.1. Each time a LC-MS instrument is used for casework, an entry shall be documented in an instrument logbook. For any sample prepared for analysis related to casework an experimental material control shall be analyzed by the same method to determine potential contamination sources resulting from solvents, lab ware, concentration steps, or other sources. All unknown samples, material controls, and reference standard analyses shall be preceded by a solvent blank, which should be devoid of responses from the analyte(s) of interest. Any response to a target component in a solvent blank or control sample shall be documented and addressed in the case notes.

5.5. Instrument and Periodic Maintenance

5.5.1. All instrument maintenance that is necessary for proper instrument performance (ex. - column replacement, pump oil replacement, ion source or analyzer cleaning, etc.) will be documented in an instrument maintenance log. The log will also document the symptoms and maintenance for any electrical component failures.

5.6. Requirements for Identification

5.6.1. At least four identification points are required to identify an analyte(s) of interest. One of these identification points shall be the retention time. Additional identification points can include characteristic ions and information from other detectors. The types of identification points used are specific to a method and/or analyte, but the total number of identification points shall not be less than four. The following list outlines the requirements for each type of identification point:

5.6.1.1. Retention Time

Comparison of sample retention times to the reference standard retention times is required for the retention time to be used as a point of identification. The retention time of each targeted analyte will count as one point of identification if it agrees with that of the standard within 5% (or less, as determined by the method validation). This is calculated as follows:

$$\frac{(\text{reference retention time} - \text{sample retention time})}{\text{sample retention time}} = -0.05 \text{ to } 0.05$$

The peak for the targeted component in the total ion chromatogram (TIC) or extracted ion chromatogram (EIC) shall have a signal-to-noise ratio (S/N) of at least 3:1.



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5.6.1.2. Characteristic Ions

Characteristic ions can include the molecular ion or reproducible fragment ions and adducts. These ions should have a relative abundance exceeding approximately 10%, except for isotopes. For LC-MS/MS analysis, characteristic ions should have a relative abundance exceeding approximately 3%, except for isotopes. If possible, the molecular ion or pseudo-molecular ion should be selected as a characteristic ion. Comparison of sample mass spectra (including relative abundance and S/N) to the reference standard mass spectra is required for any characteristic ions used as points of identification. An extracted ion profile of a characteristic ion shall have a S/N threshold of at least 3:1. The mass spectrum from the unknown sample(s) should compare well to the spectrum obtained from the standard and there should be a general correspondence between the relative abundance for the unknown and standard (for example, within 10% of each ion's relative abundance). Any significant inconsistencies between the sample and the reference standard spectrum must be explained or an identification cannot be made. For example, any contamination from filters and solvent may be explained in the case jacket and not prevent an identification. Subtraction of background spectra is permissible and may be required to remove noise.

Each characteristic ion that meets these requirements will count as one point of identification. Therefore, with the retention time as one point of identification, only three characteristic ions would be needed for four total points of identification. If the mass spectrometer is capable of accurate mass measurements, two identification points may be assigned for each characteristic ion if the mass error is less than or equal to 5 ppm.

5.6.1.3. Information from Other Detectors

Some LC-MS instruments also possess a UV detector, which can provide a point of identification. Additional identification points can also be attained through alternate ionization and/or chromatographic methods. Another instrumental/analytical technique can also be utilized.

5.7. Sources of Error

- 5.7.1. A possible source of error can result from impurities in solvents and/or carryover from prior analyses, which can be eliminated through the analysis of standards, blanks, and material controls. Environmental conditions in which instruments, especially time-of-flight mass analyzers, operate could cause gradual de-tuning/calibrating of the instrument. This can be minimized by tuning/calibrating according to the manufacturer's recommendations. The temperature and sample



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concentration can also have an effect on the retention time and chromatographic separation. This can be addressed by keeping the column compartment at a constant temperature and by diluting samples prior to analysis. Additional sources of error include co-eluting compounds and ionization suppression, which can obscure a response. Errors due to co-eluting compounds are minimized since the liquid chromatograph is connected to a mass spectrometer and ionization suppression is minimized by proper sample preparation.

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