



TE07 - Raman Spectrometer ATF-LS-TE07 Raman Spectrometer	Published Online: March 2018
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I. **Scope:**

Raman spectroscopy is a technique that has been validated for rapid identification of unknown chemicals. When a laser beam is focused on a sample, light is scattered from a molecule and most photons are elastically scattered. However, a small fraction of light is scattered at optical frequencies different from, and usually lower than, the frequency of the incident photons. The process leading to this inelastic scatter is termed the Raman effect. Raman scattering can occur with a change in vibrational, rotational or electronic energy of a molecule. The frequencies and intensities of the Raman scattered photons relate to the conformation and electronic states of the probed molecule. Thus, the Raman spectrum of a material can be used as a unique chemical signature of the material.

The Raman Spectrometer is designed for field and laboratory use to provide rapid identification and comparison of unknown solids, liquids and mixtures by comparison with known Raman spectra.

The analysis is non-destructive, and is an effective complement to FTIR. Raman spectroscopy and FTIR are both based on molecular vibrations. FTIR measures the absorption of infrared light by the sampled material. Water is a strong infrared absorber and often masks the presence of other compounds. Raman on the other hand measures the light that is radiated by the unknown substance and is not troubled by water, thus allowing analysis of aqueous solutions.

II. **References:**

Operations manual/ Quick Reference Guide for the Ahura FirstDefender and the Thermo Scientific FirstDefender RMX

Ahura Scientific FirstDefender, The Science Training Session V2.5.2, Prepared by Dr. Javier Santillán, Application Scientist

Army Validation Studies for the Ahura FirstDefender:

http://www.ecbc.army.mil/downloads/reports/TR_528.pdf

http://www.ecbc.army.mil/downloads/reports/TR-461_Matthews.pdf

Instrumental Methods of Analysis, by Willard, Merritt and Dean, published by D. Van Nostrand Company, Inc.

Raman Technical Resources, Raman Tutorial, Kaiser Optical Systems, Inc.

<http://www.kosi.com/raman/resources/tutorial>



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Raman Spectroscopy, On-line chemistry class, University of Colorado, Denver
<http://carbon.cudenver.edu/public/chemistry/classes/chem4538/raman.htm>

Analysis of a Suspect Explosive Component: Hydrogen Peroxide in Hair Coloring Developer, Edward G. Bartick, Rena A. Merrill and Kelly H. Mount, in: Forensic Science Communications, October 2001, Volume 3, Number 4

Raman spectroscopy as a tool for long-term energetic material stability studies
David S. Moore, Kien-Yin Lee, in Journal of Raman Spectroscopy, Volume 38
Issue 9, Pages 1221 - 1224

Validation:

Raman Spectroscopy is a well known and scientifically accepted method for the identification, analysis and comparison of many types of trace evidence. Relevant examples of the broad nature of the method and related literature can be found in Section II (References).

III. Safety Precautions:

Hand-held Raman Units:

- See Appendix I for special safety considerations when using the hand-held units.

IV. Apparatus/Reagents:

- Hand-Held Raman Spectrometer
- Factory-installed software and library of commercial and industrial compounds, narcotics, toxic industrial chemicals and toxic substances.
- User created libraries

Logbooks will be kept which document the performance checks as well as maintenance on the instrument.

Checking the Hand-held Instrument Performance:

The Ahura FirstDefenderXL and Thermo Scientific FirstDefender RMX spectrometers are initially calibrated at the factory, and have an ongoing internal performance check program. Results of this internal performance check will be printed on the report of each run. If the unit falls outside of the internal calibration specs, a warning icon will appear and the unit may require factory service. No user calibration option exists.



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A polystyrene standard (provided with the instrument) allows the user to perform a confidence check. This will demonstrate that the instrument is operating correctly. It is the responsibility of the user to ensure that prior to sample analysis, a confidence check has been performed and evaluated within the last thirty (30) days. The data from the confidence checks will be retained for a period of at least five (5) years or one ASCLD-LAB International inspection cycle.

V. Procedures:

Preparing and Running Samples on the Hand-held Raman Units:

After obtaining a satisfactory internal check and confidence check as outlined above, the sample of interest may be analyzed. Sampling and preparation will vary depending on the type of sample to be analyzed. The hand-held units have three different methods for scanning samples. Samples may be run using a vial inserted into the internal sampling chamber of the instrument or they may be run externally with or without the nose cone attachment.

The following tips may help in getting a better scan:

- While scanning, use of a ring stand may help to stabilize the unit.
- The Signal Strength option may be used to adjust the unit into the position which results in the greatest intensity reading.
- In case of non-uniformity of samples, it may be desirable to sample the material in several different areas. When using a vial, the sample vial may be rotated during the scan so the laser is able to detect each type of substance that may be present.
- The unit generally displays results in thirty (30) seconds or less. If the unit displays an estimated scan time of several minutes, one of the following problems may be occurring:
 - The focal point is not correctly positioned or the wrong scanning technique is being employed.
 - The substance being scanned has a very weak molecular signal or is highly fluorescent (examples: table salt, ammonia, muriatic acid, flour, baking powder, brake fluid, coffee, soda).
 - Ambient light is increasing fluorescence and should be minimized.
- For specific instructions on using the software and taking a sample spectra, the operator should consult the manufacturer's manual or quick reference guide.

Internal Scanning of Samples

This method may be used to analyze liquids, powders, or pastes. The sample of interest should be placed in the sample vial (do not cap vial) with enough sample



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to ensure that the focal point of the laser is correctly positioned on the sample pile when placed in the chamber at a 45 degree angle. When sampling a paste, the sample must be in contact with the side of the vial that the laser light contacts first. When sampling materials from home-made explosives (HME's), this amount should not exceed but 300 mg (~pea size) or 300 uL, (~5 drops).

Scanning of Samples without the Nose Cone Attachment

This method is best employed when sampling a substance that is inside a thick walled translucent container (more than 1/8" thick). The focal point of the laser is approximately 3/4" away from the nose of the instrument which will provide enough distance for the focal point of the laser to be positioned beyond the container walls.

Scanning of Samples with the Nose Cone Attachment

This method is best employed when sampling a substance that is inside a thin walled translucent container (1/8" or less in thickness) by touching the nose cone to the side of the container during the scan. Puddled liquids, piles of powders, and larger solid substances may also be analyzed using this method by holding the tip of the nose cone in contact with the liquid, powder, or solid. Thinly spread liquids or films may be collected onto a cotton swab (the swab must be wet not just damp) and analyzed by touching the nose cone to the cotton swab. Be sure not to place samples on paper towels or similar cellulosic material to avoid heating of those materials.

Interpretation of Scan Results for Hand-held Raman Units:

After scanning a sample, the unit will display one of the following result screens:

- Positive Match Found – This means that the data matches only one library item.
- Positive Matches Found – This means that the data matches two or more library items meaning the substance could be any one of them.
- Mixture – Meaning a single positive match for the substance could not be found and a mixture of library items match the data. Mixture identification is a complex analysis and is more susceptible to false positives than the “one positive match” or “multiple positive matches” results. Care should be taken when interpreting these results.
- No Match Found – This means the substance being tested did not match anything in the library, including mixtures of the library substances. In this case, the unit will list any library items that are similar to the substance in question. Care should be taken when interpreting this type of result as it does not mean that the listed substances are present, only that the data resembles them in some respects. A repeat scan of the sample using a different sampling method may provide additional information.

Reports of selected scans may be saved onto a memory card and transferred to computer using the CF card reader. Printouts of these reports should be included in the case file.



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VI. Quality Assurance/Quality Control:

Through regular confidence checks using the known standards, and upkeep of instrument logbooks, the quality of the Raman method is assured. The instrument confidence check data and maintenance log should be kept in electronic format or in a logbook.

There is no known error rate for this type of examination.

Appendix I:

Safety Considerations for Hand-held Raman Units:

Portable Optical Analysis 46 Jonspin Road Wilmington, MA (978) 657-5555 www.thermoscientific.com/ahura
01887 (978) 657-5921 fax

Technical Service Bulletin TSB0002

Title: Update on Best Practices for Raman Measurement of HMEs April 15, 2011

SUMMARY

This bulletin summarizes the recommended best practices for the safe measurement of home-made explosives (HMEs) and suspect HMEs using Raman spectroscopy. This list is a compilation of techniques developed by bomb technicians, responders, and other experts. The techniques and recommendations are based on field experiences and the outcome of various tests and evaluations. Using Raman spectroscopy, our FirstDefender product line aids military and local bomb squads in identifying explosives and their chemical precursors. FirstDefender products are widely used each day safely in these demanding environments and situations.

The physics of Raman spectroscopy requires that a laser light source be shined on the material being tested. Shining light onto a material can cause localized heating. This heating can cause the ignition of some highly unstable samples or darker colored materials. **Dark colored materials are most susceptible to ignition by a Raman laser. Some lighter colored sensitive/unstable materials (e.g. Nitrocellulose and Mercury Fulminate) or dirty materials containing dark flecks can pose ignition risks as well.** The analysis of home-made explosives is inherently unsafe. The recommendations compiled here are the latest techniques on how best to safely measure questionable samples in a variety of environments using Raman spectroscopy.

PRODUCT LINES IMPACTED

Raman platforms including FirstDefender, FirstDefender RM and FirstDefender RMX.

The following Safety Guidelines should always be used when suspected energetic materials are present:



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- In all cases, the **safest practice** is to **isolate** and measure a **small amount** of material in an **uncapped vial** and to use the **low laser power** setting on the instrument. Also, **use the scan delay feature** when the material color or purity is questionable. See the appropriate sections of the User Documentation for using the scan delay feature.
- If vial sampling is not appropriate, it is recommended that a **small isolated pile** (<300 mg, ~pea size, or <300 uL, ~5 drops) be measured in point-and-shoot mode using the **scan delay feature** to provide operator distance.
- A small pile of material is easier to measure, faster, and safer than a thin layer of material on top of another surface.
- It is **NOT** advisable to measure suspected energetic materials on filter paper, similar cotton bases (e.g., paper towel) as heating of those materials may occur.
- If sampling is not appropriate, and point-and-shoot measurements on large quantities of material or materials in sealed containers must be conducted, it is **CRITICAL** that **the scan delay feature** be used to provide a safe operator distance.
- Wear personal protective gear as appropriate for not only the blast hazard but the possible **chemical hazard** (e.g. skin chemical exposure). A medical review should be considered after exposures to hazardous chemicals.
- Do **NOT** conduct library builds on suspected energetic materials without appropriate precautions.
- Library building scans the materials for a long period of time.
- Scanning the same location of a sample increases the ignition hazard.
www.thermoscientific.com/ahura

Eye Safety Guidelines:

- Never point the laser at people and maintain the appropriate distance between the laser aperture (nose) and the operator's eyes. (14" for AhuraFD, 19" for FirstDefender RM, and 17.25" FirstDefender RMX)
- Stay behind the incorporated laser eye shield when performing a scan



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- For any questions, details, or additional support, please contact us 24 hrs / 7 days at 800-374-1992 (U.S. domestic toll-free), +1-978-642-1100 (International), email to support@chemid.thermofisher.com or visit the Customer Support website www.thermoscientific.com/chemid-support