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# IRAQI JOURNAL OF APPLIED PHYSICS

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[2] M. Barnes, "Stresses in Solenoids", *J. Appl. Phys.*, 48(5) (2001) 2000–2008.
[3] J. Jones, "Contact Mechanics", Cambridge University Press (Cambridge, UK) (2000), Ch.6, p.56.
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[7] C.Y. Tung, "Evaporative Heat Transfer in the Contact Line of a Mixture", Ph.D. thesis, Rensselaer Polytechnic Institute, Troy, NY (1982).

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# Olive Oil Analysis Using FT-NIR Spectroscopy

*Sponsored by Galaxy Scientific Inc* Apr 17 2019

**Olive oil consumption is continuing to grow globally, making it a high-value foodstuff. Regardless of what position in the olive oil industry a business sits it is important for a business' reputation that the quality and authenticity of their oil is guaranteed. In addition, it is also important that oil processing is carried out efficiently to increase profitability.**

## Principles of Near-Infrared Spectroscopy

NIR (Near-infrared spectroscopy) is becoming an invaluable tool for the food industry, allowing businesses to rapidly carry out process monitoring, quality control and to improve efficiencies. The advanced Fourier Transform Near-Infrared (FT-NIR) Spectrometers provided by Galaxy Scientific are rugged, compact and low-maintenance whilst providing excellent performance.

NIR spectrometers work by exposing the sample to an NIR laser, which either passes through the sample or reflects off it, following which the remaining NIR laser signal is measured. The wavelength profile of the NIR laser is changed by its interaction with the sample and this change depends on the sample's chemical composition. NIR spectroscopy allows all of the different chemical constituents of a sample to be identified using just one measurement that can be carried out in under a minute.

The technique is highly useful because samples do not need to prepared for measurement, they must simply be placed in a cup or vial before introduction to the spectrometer. In addition, the spectrometers are straightforward to use and can be operated with just a touch of the button, meaning non-experts can carry out the analysis.

Businesses that adopt FT-NIR into their processes no longer need to use expensive labs or third parties to carry out analysis of their olive pastes, pomace or oils and can instead carry out a fast, economic and precise analysis in-house.

## FT-NIR Spectrometers

The FT-NIR spectrometers provided by Galaxy Scientific allow producers to immediately chemically analyze their input products, such as olive paste, and output products, such as pomace and olive oil. This immediate feedback means producers can adjust their extraction processes to improve the quality of their product.

FT-NIR can be used to determine the concentration of oil and moisture in olive pastes, allowing harvests to be optimized to reduce product loss and increase profitability.

FT-NIR can also be used to assess the quality of olive oil being produced. Important quality parameters, such as the oil's -values (K232, K270, delta K, which are important for extra virgin classification), the oxidation state, FFA (free fatty acids) content and acidity, can be measured. In addition, nutritional content and other important parameters can also be determined to help increase profitability when selling to other manufacturers.

The residual oil content of the waste pomace can also be assessed, allowing manufacturers to find out how much oil is not being extracted successfully.

## QuasIR™ Series—A New Generaion of FT-NIR Systems from Galaxy Scientific

The FT-NIR systems from Galaxy Scientific have been designed by expert scientists with

the requirements of the olive oil industry in mind. The instrument is easily operated via a simple point-and-click interface. Single measurements can be carried out at the touch of a button using the system's Spectral Sage EZ software. Custom reports can be produced with batch and lot IDs included for later reference.

The QuasIR™ 4000 is can be used for both at-line and on-line measurements, being able to analyze paste and pomaces in reflection mode, and oils in transmission mode.

The QuasIR™ 2000 can be used to automate measurements, providing continuous, dynamic data, making it perfect for use in high-volume production environments. Sample

probes can be inserted directly into the production pipeline, with the IR data sent to the instrument via fiber optic cables. Reflection probes can be used to analyze solid process materials, such as pomace and pastes, whereas a transmission probe can be used to analyze olive oil.

Galaxy Scientific's new multiplexer means up to ten sampling points can be measured using a single instrument. This lowers the cost of running a spectroscopic analysis and improves the manufacturers ROI on the instrument.

## Benefits of Using FT-IR for Olive Oil Analysis

- A fast and accurate method for the analysis of olive oil, pomace and paste
- Requires no sample preparation or consumables, resulting in a low cost of ownership
- Perfect for quality assurance and control
- Spectrometers can be used to measure several parameters of interest at once
- Can be used to take dynamic measurements both in-process and off-line

## Parameters for Olive Oil Analysis

- Esters
- Extinction Coefficients
- Peroxide Index
- Fatty Acids
- Linoleic, Oleic and Palmetic Acid
- Adulterant screening
- Moisture and volatile matter

**Lab or Online Measurements of:**

- Oil
- Pomace
- Paste


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# Using Energy Dispersive Spectroscopy to Analyze Gun Shot Residue

*Sponsored by EDAX Inc.* Apr 4 2019

**Scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS) is an industry-approved technology for the identification and analysis of typical Gun Shot Residue (GSR) primer particles, thanks to its speed and ability to analyze individual particles.**

Systems, which encompass a large amount of analysis area utilizing built-in SEM/EDS controls, are developed for automated analysis of GSR particles with less user intervention. Software and hardware components are employed to identify particles of choice and then further organize them into classes of interest that are characteristic of GSR.

When a GSR system is specifically developed to increase the data collection efficiency, more sampling area can be covered in a reduced amount of time, which results in higher productivity and a greater possibility for detecting even the smallest GSR particles present in a sample.

## Analytical Requirements

The image detection of each GSR particle and the collection of spectral data from each particle from a whole set of stubs in the sample are two major aspects of a run. It is essential to precisely and efficiently acquire key parameters and details like particle morphology, stage location, classification matching, and chemistry.

Several GSR samples can contain hundreds or even thousands of particles of interest, thus even small reductions in collection time per particle, without affecting the classification accuracy, will be advantageous to the total throughput and productivity of a GSR analysis run. Moreover, advanced follow-up analyses of classified GSR particles validate the match and improve the overall confidence in results.

## EDS Silicon Drift Detector (SDD) Collection Parameters and Optimization

A number of factors are vital in the total EDS data collection process, such as:

1. Input count rate
2. Output count rate
3. Scan speed or collection time

Higher count rates and more rapid collection speeds will result in an increased speed of data collection. It is essential to take each aspect into consideration when expressing the detector's collection speed for the analysis.

1. Input count rate (ICR) refers to the number of X-rays that strike the detector and is given in counts per second (CPS). This factor is a function of the sample, SEM and detector geometry (which means some low atomic number samples do not generate a high number of CPS, while metallic particles generate a high number of CPS). The SEM operation, kV, and beam current have two key variables. The widely established practices for GSR analysis use a beam voltage of 20–25 kV, which is extremely suitable for the production of high CPS. The SEM's beam current can be easily modified to attain high ICR, while still retaining the needed quality to detect and image even the tiniest required submicron GSR particles. Finally, the detector geometry considerations will have an effect on the input count rate; however, usually, any size detector will readily obtain the highest required count rates on the basis of the sample and SEM conditions. Yet another factor is the window material. Although windowless detectors are not appropriate for GSR due to contamination problems[1], developments in thinner window materials enable improved input count rate, while still guaranteeing SDD protection and cleanliness.
2. Output count rate is a very vital factor in the EDS data collection process. It is a function of how efficiently the electronics of detector turn the raw input count rates into the useable X-ray counts in a spectrum. For data characterization, high input count rates can be converted with low dead time into usable counts but only with a fast processing time, typically below 1 µs. Additionally, the detector needs to maintain a high-quality resolution at faster processing time. Instruments having a resolution of about 130 eV for the Mn Kα peak will more unambiguously resolve close peaks, for example, the Ca Kα and Sb Lα1 peaks.[1]
3. When the SDD can be optimized for count rates, spectral collection time becomes a secondary function. Collection times as rapid as 1 second or better provide a quality GSR classification and analysis with a detector that delivers high efficiency, as well as quality resolution. For instance, an SDD, which obtains more than 100K CPS with less than 30% dead time, while still retaining a resolution of 130 eV or more may be employed. This will guarantee an analytical method where countless numbers of particles from several samples and stubs can be analyzed in fewer

hours when compared to the conventional overnight runs. Shorter run times provide users the opportunity to manually verify the results several times per day, rather than just once as with an overnight run.

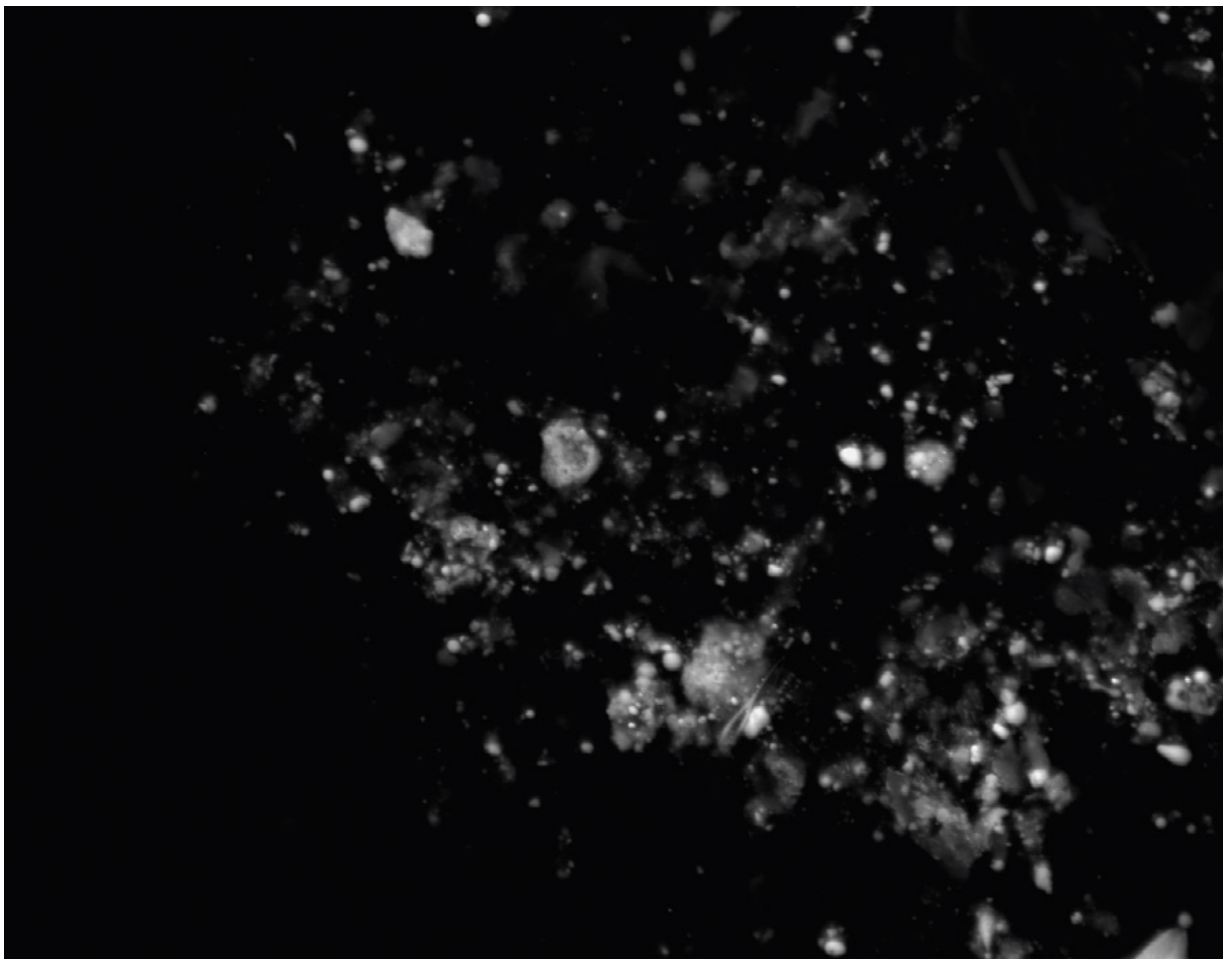

*Figure 1 shows a 500X magnification of a sample area of a GSR stub. At 2048 x 1600 pixel resolution spectra from particles as small as 0.25 µm can be collected.*

Particle Analyzed/Found 308/308

| Class | Class name | Analyzed | Total | Part/sq.mm | Area Fraction (%) |
|---|---|---|---|---|---|
| 1 3 | Comp | 94 | 94 | 1835.94 | 0.9888 |
| 3 2 | Comp Sb Pb | 8 | 8 | 156.25 | 0.033 |
| 4 2 | Comp Ba Pb | 153 | 153 | 2988.28 | 2.3 |
| 6 1 | Comp Ba Pb | 33 | 33 | 644.53 | 0.307 |
| 7 1 | Comp Pb | 15 | 15 | 292.97 | 0.062 |
| 14 | Fe | 2 | 2 | 39.06 | 0.253 |
| | Unclassified | 3 | 3 | 58.59 | 0.01 |

*Figure 2 shows the analyzed results of the 308 particles found in this single field of view.*

*At one second collection time, this data was collected in about 5 minutes.*

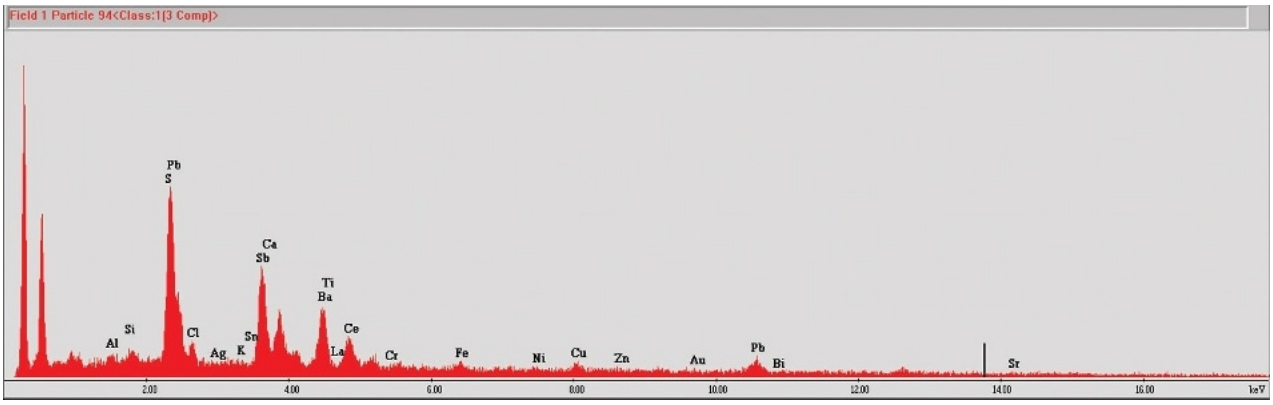


*Figure 3 shows a spectrum collected in 1 second. Octane Elite yielded high input count rate (117 KCPS) and low dead time (26%) with a high output count rate of 86 KCPS with a 130 eV quality resolution, so there is high confidence in the statistical accuracy of the classified 3-component GSR particle.*

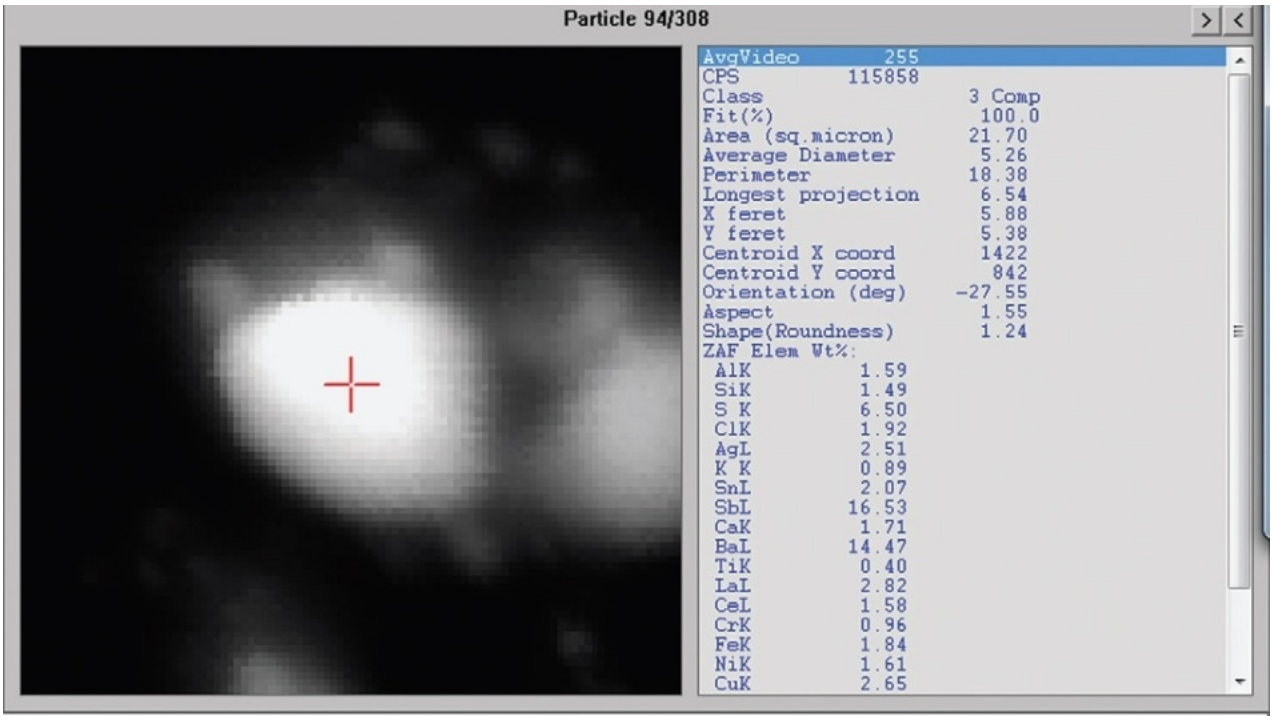


*Figure 4 shows a zoomed-in area of the 3 component particle that corresponds with the spectrum in Figure 3.*

## EDAX Octane Elite Series

The EDAX Octane Elite EDS System has numerous prominent design characteristics which make it ideal for GSR applications. The most noteworthy benefit is the innovative Silicon Nitride window material, which is designed to be almost 10 times thinner than

conventional polymer windows, while still maintaining the detector integrity. This will generate higher input count rates to the detector. Moreover, Octane Elite electronics are one of the fastest available in the market with processing times less than 0.2 µs, which guarantees the efficient and reliable conversion of input count rates into output count rates.

Additionally, the Octane Elite electronics provide resolution stability and will guarantee the detector resolution quality to separate close peaks and offer precise GSR classification solutions. In a nutshell, the most recent design technologies of the Octane Elite detectors take GSR analysis to a whole new level.

## Reference

1. *Guide for Primer Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive X-Ray, Spectrometry 11/29/11*

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## Primary Activity

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# A Guide to the Prisms Used in Spectroscopy

*By Rebecca Ingle* Apr 23 2019

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**Prisms are commonly used in spectroscopy for dispersing or controlling the direction or polarisation of light. This can be helpful for splitting the incoming light into its wavelength components to maximize the information recorded with a multi-channel detector, such as a CCD camera, or performing polarization-controlled experiments. Using different polarisations of light can help assign certain spectral features, such as the nature of vibrational modes in a Raman experiment.**

There are a wealth of prisms available with different properties and specifications for all of these applications, including the ability to either change or retain the direction of the incoming light. Some, such as Dove prisms, will not deflect the beam or introduce any refraction but will invert the image top to bottom. This makes them useful for changing the direction of linearly polarised light without affecting the beam steering.

## Prisms for Beam Steering

Beam steering is one of the most common uses of prisms in spectroscopy. While mirrors tend to be cheaper and result in fewer transmission losses, as with a prism the beam must pass through the prism's material, for very compact experiment with very accurate alignment, prisms can be preferable. The error on mirror alignment is always doubled due to the Law of Reflection and, while mirrors allow for more alignment flexibility, it can be difficult to set them up and adjust them to get precise reflection angles.

Commonly used prisms for beam steering include right angle prisms, that can be used to reflect a beam either 90◦ or 180◦, depending on the prism's orientation. Wedge prisms are highly versatile and can deflect the beam any number of degrees while also helping to correct for small ellipticities in the output beam shapes from diodes. Other prisms used for steering include pentaprisms or Pellin-Broca prisms. Pellin-Broca also has the advantage that the angle of rotation of the prisms will deviate only a specific wavelength of the incident light by 90◦ and can, therefore, be used for separating wavelengths following a process like frequency doubling, where some of the residual fundamental may remain colinear with the output beam.

## Dispersing Light

There are a number of different prisms designs that are suitable for maximum dispersion of the incident light. Probably the most iconic of these is the triangular prism, which resembles an equilateral triangle. Dispersion in the material occurs as the different wavelength travel at different speeds changing the path accordingly. Changing the prism design can also influence the amount of dispersion, for example, triangular prisms tend to be more expensive to manufacture, so Littrow prisms were often used in their place and can be useful where a narrower range of dispersion is required.

For dispersion of light in spectrometers for detection, the increased affordability of diffraction gratings means that prisms tend to be used only for applications where the poor transmission of gratings causes issues. However, control of the dispersion of light with prisms is of utmost importance in ultrafast spectroscopy as it is essential for keeping laser pulses temporally compressed and maintaining good time resolution. Here, triangular prisms are often used in opposing pairs to compensate for the dispersion each individual prism applies to the light.

## Prism Materials

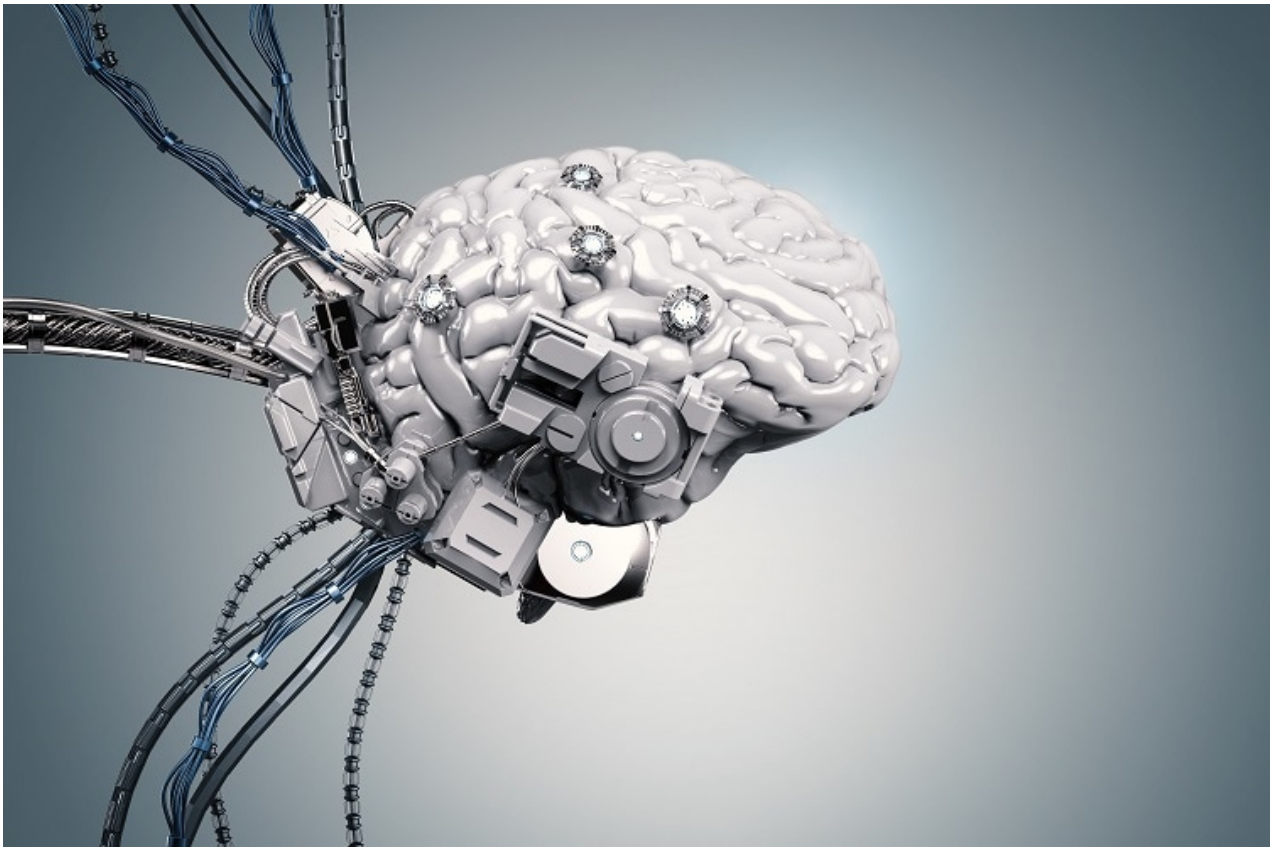
As spectroscopy makes use of most of the regions of the electromagnetic spectrum, the materials of a prism need to have relatively high transmission in the wavelength region being used. Fused silica is a popular choice for the UV and visible range and the addition of anti-reflective coatings can help enhance transmission for specific wavelength regions as well. BK7 is a cheaper alternative in the visible to infrared range, though, for longer IR wavelengths zinc selenide, germanium and silicon are necessary.

## Versatile Optics

Prisms can be used for a number of purposes in spectroscopy, including beam steering, dispersion and polarisation control. The number of different prism geometries and coatings, as well as their compact footprint, means they are also easy to integrate into a range of devices and experiments.

# Application to Robot Control Using Brain Function Measurement by Near-Infrared Spectroscopy

*By Benedette Cuffari, M.Sc.* Apr 3 2019



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**In recent years, research on the brain, computer interface (BCI), has been conducted quite extensively. While some of the earlier BCI studies utilized electroencephalogram (EEG) data, other studies have reported the use of near infrared spectroscopy (NIRS) as a useful and informative method.**

Because NIRS is a non-invasive technique and only requires the use of small-scale instruments, it can be incorporated into a wide variety of BCI applications, especially when compared to those based on EEGs.

## Challenges Faced by Previous BCI Studies

Previous BCI studies required the researchers to be extensively trained to ensure that they were capable of handling BCI instruments in a skillful manner. More specifically, the individual handling BCI was required to control his or her brain signals in an efficient manner.

For example, if the person using the BCI's brain signal had limited motor control of their arms or legs, the BCI would not work effectively. Therefore, the person in which the BCI was intended for was obligated to undergo extensive and grueling sessions of complicated training prior to handling the BCI.

To alleviate these challenges, scientists have recently been successful in their development of more practical BCIs that were simple to use and operate through the performance of simple actions, such as the movement of the individual's arms or legs.

## Using Brain Signals on Pyramidal Area to Develop NIRS-Based BCI

Researchers at the Nagaoka University in Japan have published their research on NIRS-based BCI that has several potential applications to control the 'on' or 'off' functions of the BCI. This research can be found in the Proceedings of the 29$^{th}$ Annual International Conference of the IEEE EMBS, which took place in Lyon, France.

It is common knowledge in neuroscience that the movement of the arms or legs results in activation around the pyramidal area of the central nervous system (CNS). Tsubone's group at the Nagaoka University have successfully estimated the start and end timing of a person's tapping movement by utilizing the NIRS signal around the pyramidal area.

Using NIRS, this group of researchers measured the hemoglobin (Hb) signals during an individual's movement. The analysis of these signals suggested that that movement of the subjects' arms or legs resulted in an increase of oxy-hemoglobin (oxy-Hb) and total hemoglobin (total Hb); however, a decrease in deoxy-hemoglobin (deoxy-Hb) levels were found within the motor cortex of the brain.

Interestingly, the Hb signals in the motor cortex depended less on the frequency of the movement, and instead relied more heavily on the intensity of the muscle activity, thereby signifying that maximum tapping effort would result in a larger activation of the Hb signals.

By collecting data from several subjects that were performing motor activity, which

involved tapping their right hand on the desk, and analyzing the cerebral blood flow and Hb levels at different times, movement amplitudes and speeds, Tsubone's team tried to estimate the start and end timing of the tapping movement throughout the neural network.

An OMM-3000 NIRS system was also used in this study. After the probes are arranged at specific regions, near IR light was focused on the scalp from incident probes, whereas detector probes were used to measure the light as it passed through the cerebral cortex in order to estimate the Hb levels.

The start and end timing of the movement of the arms by the human subjects were estimated by classifying the states into three types, of which included an increase in Hb, a decrease in Hb and no variation of Hb.

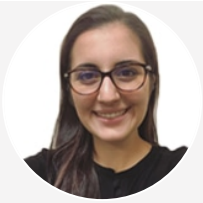
Each of these types of responses were used to design a learned network based on the data acquired throughout the study. Using this method, the researchers were able to estimate the 'on' or 'off' control of the BCI with a success rate of more than 70%.

## Conclusion

The research discussed here demonstrated that learned networks can be used to estimate both the start and end timing of muscle movement by individuals. With this information, researchers can design effective BCIs capable of detecting simple human movements which can ultimately be applied to a wide range of biomedical purposes.

## Reference and Further Reading

1. Tsubone, T., Muroga, T., & Wada, Y. (2007). Application to robot control using brain function measurement by near-infared spectroscopy. *Iee Xplore.* DOI: 10.1109/IEMBS.2007.435348.



**Written by**

## Benedette Cuffari

After completing her Bachelor of Science in Toxicology with two minors in Spanish and Chemistry in 2016, Benedette continued her studies to complete her Master of Science in Toxicology in May of 2018. During graduate school, Benedette investigated the dermatotoxicity of mechlorethamine and bendamustine, which are two nitrogen mustard alkylating agents that are currently used in anticancer therapy.

# Elemental Analysis of Honey to Detect Pollution

By Lakshmi Supriya, PhD. Apr 2 2019

*ImageCredit:Shutterstock/kosolovskyy*

**The pollution of the environment by humans has been exponentially growing. Factories and vehicles spewing out noxious fumes poison the air that we breathe. Liquid waste and discharges from factories and communities into water bodies pollutes rivers, lakes, and groundwater.**

When food is grown using polluted groundwater for irrigation or where the air is contaminated, there is a high likelihood that the grown fruits, vegetables, grains, and crops are also polluted.

Among the several methods of monitoring environmental pollution, analyzing foods is one method of keeping a tab on pollution. The analyses performed may include chemical analysis to detect chemical contaminants such as heavy metals, and biological analysis to detect biological pollutants such as harmful microorganisms. Honey is one food that can be analyzed to detect pollution.

# Detecting Pollution Using Honey

Bees produce honey by converting the nectar that they take from flowers. They also drink water and breathe in air, which may be polluted. Since bees generally fly around and visit a large number of flowers in a small radius from their hive in search of nectar, they can be used to monitor pollution over local, and small areas.

Using chemical analysis techniques, various hazardous compounds can be detected, giving an indication of the environmental pollution in the area. Bees have been used for a long time in monitoring pollution. For example, a study published in 1975 reported elemental analysis of honey to detect whether honey produced near highways or industrial areas was polluted.[1]

Using spark source mass spectrometry, the authors found that the honey collected from nearby highways contained elements such as barium, nickel, and silicon, which are generally known to be produced by traffic.

Recently, several other methods of elemental analysis have been used to detect pollutants in honey. Most methods usually detect heavy metals. Some heavy metals like iron, manganese, and nickel are important for human growth in low concentrations. However, several metals such as lead, cadmium, chromium, and mercury are extremely toxic, even at low concentrations in the body.

Metals in honey can be present if the hives are located close to factories, highways, mines, or other industrial areas. One study used inductively coupled plasma-optical emission spectrometry (ICP-OES) to detect the presence of metals in honey.[2]

The samples were treated first to remove most of the organic matrix using solid phase extraction or wet digestion. The ICP-OES uses a plasma to convert the elements to their respective atoms and ions that emit characteristic radiations which are then used to identify them.

Another study examined trace elements present in honey produced on reclaimed uranium mining land in Texas, USA.[3] Honey samples obtained from the reclaimed land were analyzed by the use of neutron activation analysis. This technique uses minimal sample preparation and the samples are irradiated with neutrons.

This produces radioactive isotopes and the elements can be identified by their

characteristic decay signature. The study found that the heavy metal content of the honey samples was comparable to those found in commercially accepted honey.

Apart from heavy metals, analysis of honey can also be used to detect environmental contamination by pesticides and other persistent organic pollutants (POP).

Researchers in Italy used gas chromatography-mass spectrometry (GC-MS) to detect pollutants in honey produced in three different areas of Italy.[4] Solvent extraction was first used to extract the organics, which were then analyzed using the GC-MS.

Honey can also be used to detect pollution by radioactive materials. A team of researchers analyzed honey produced in Poland to see if it had been contaminated by radioactive materials after the Fukushima nuclear plant disaster in Japan.[5]

Most radioactive materials produce gamma rays that are characteristic of each material. So, the researchers used gamma-ray spectrometry to determine that the amount of radioactive materials did not increase in Polish honey after the disaster.

## Conclusion

Honey from bees can be a powerful indicator of environmental pollution, particularly when the samples are taken in a small area close to the hives.

However, an important factor when analyzing the presence of pollutants in honey is to understand how the plants uptake the pollutants when the bees take nectar from them, and which parts of the plant body the pollutants are concentrated in.

In addition, it is important to ensure that the analytical techniques used to detect pollutants are performed carefully once the applicability of the technique has been verified.

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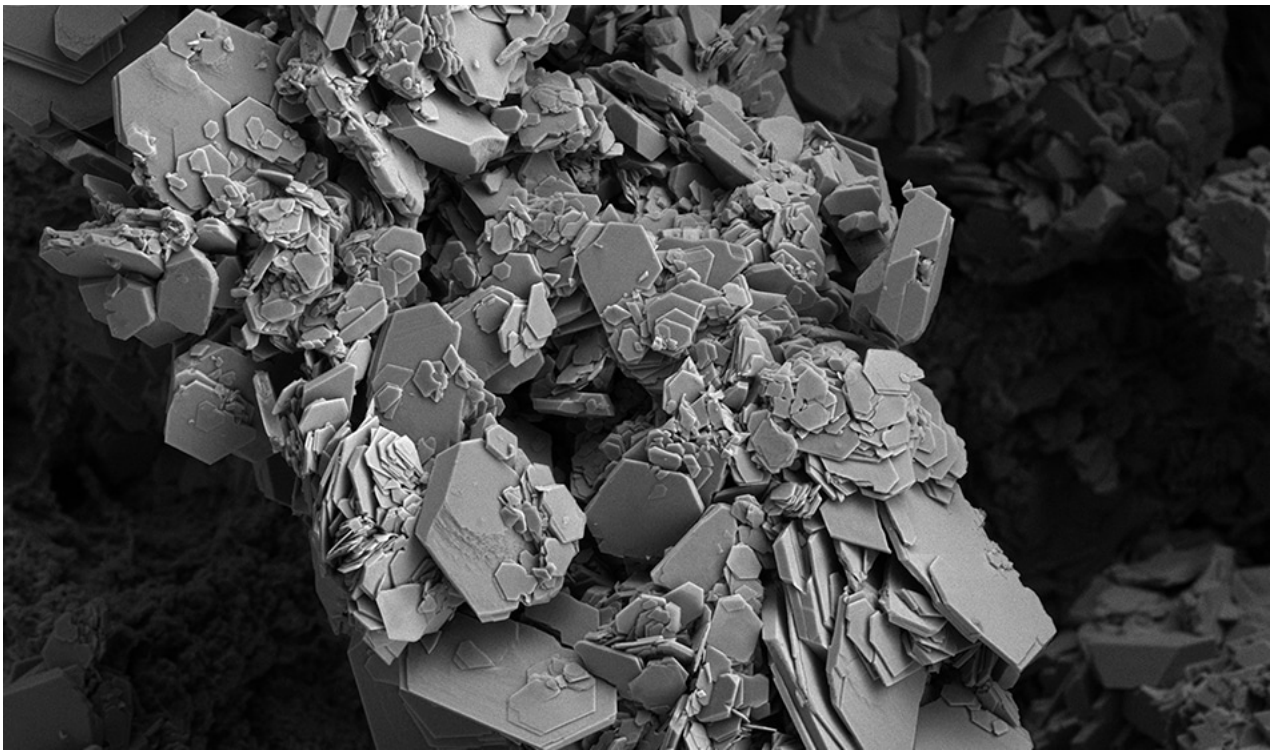
# Thin Film Elemental Analysis for Mineral Characterization

By Benedette Cuffari, M.Sc. Feb 4 2019

**Within the microelectronics, optics, photovoltaics, coatings and glass industries, researchers often require details on a wide range of thin film material properties; particularly the elemental composition of these products. When presented with this information, researchers and engineers in these industries can accurately choose the most appropriate coatings, modified surfaces and supporting materials for their products.**



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## Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectroscopy (EDS)

Scanning electron microscopy (SEM) is a well-known tool that engineers, scientists, tribologists, and lubricant engineers often utilize to analyze the morphology, defects and wear behavior of certain materials.

The combination of SEM with energy-dispersive X-ray spectroscopy (EDS) further

enhances the analytical capabilities by providing users with a more powerful method of obtaining information on the elemental composition in near-surface regions, as well as the morphological and topographical details of thin film samples.

Several studies have cited the sensitivity of SEM-EDS in characterizing the physical, chemical and mechanical properties of various mineral thin films. For example, a 2012 study utilized SEM-EDS to investigate the thermal stability of hard chromium nitride (CrN) thin films that were deposited on a silicon substrate. The combined SEM-EDS approach allowed the researchers to visualize an apparition of oxygen present within the CrN films. This oxygen presence was subsequently replaced by nitrogen until the chromium (III) oxide ($Cr_2O_3$) phase was formed. SEM-EDS data, therefore, provided valuable information regarding the distribution of these phases within the microstructure of the CrN films.

## X-ray Photoelectron Spectroscopy (XPS)

X-ray photoelectron spectroscopy (XPS), also referred to as electron spectroscopy for chemical analysis (ESCA), is one of the most commonly used techniques in the surface engineering and tribology industries.

In XPS, the sample surface is excited by mono-energetic aluminum (Al) X-rays, which cause the sample's surface to emit photoelectrons. The energy of the emitted photoelectrons is then analyzed to provide valuable quantitative and chemical state information on the material being studied.

When used for the elemental analysis of thin films, XPS has provided clear information on the film's stability and long-term reliability, which are important factors to consider prior to utilizing these constructed films for photovoltaic cells and thin film light emitting diode (LED) products.

## X-ray Diffraction (XRD)

When used for material characterization, X-ray diffraction (XRD) offers a non-destructive technique that does not require special sample preparation before analysis. Furthermore, XRD allows users to obtain structural information on relatively large areas of the sample without causing any irradiation damage in the process. The application of XRD for the analysis of thin films present in semiconductors, electrodes and piezoelectric products has provided useful information on the crystallite size, lattice strain, reflectivity measurements, elemental composition, relaxation, thickness, roughness, density and

pore/particle size distribution.

A 2019 study published in the *Journal of Alloys and Compounds* discussed the utilization of XRD to evaluate the conductivity effects that occurred when nanostructured Al was incorporated into zinc sulfide (ZnS) thin films. More specifically, the researchers used XRD to investigate the crystal phases of the film sample and determine how these structural characteristics and properties played a role in the optical, electrical and electrochemical properties of the ZnS thin films. Analysis of the XRD patterns revealed that Al-doping reduced the crystallite size of the ZnS samples, which thereby contributed to a reduced dislocation density and lattice strain of the ZnS films.

## Colored Picosecond Acoustic (APiC) Technique

The colored picosecond acoustic (APiC) technique utilizes ultrafast laser technology. This technique has demonstrated its ability to characterize specific properties of thin films, such as the adhesion quality present at the different interfaces between the film and substrate. APiC begins with shining a fast laser light pulse onto the sample, which is followed by the application of acoustic (hypersonic) waves in the same area. These sound waves propagate within the stack of thin films which causes an echo to be emitted and ultimately analyzed to determine the thickness and other material characteristics of the film sample.

A 2018 study recently investigated the potential use of APiC as a characterization method for thin film samples composed of metals, dielectrics, and semiconductors. In their work, ultra-high frequency acoustic waves provided useful information on material thickness, as well as the elasticity and adhesion properties of nickel (Ni) thin films deposited on a silicon (Si) substrate, without requiring any contact or destruction to occur throughout the analysis process.

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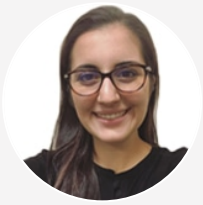


**Written by**

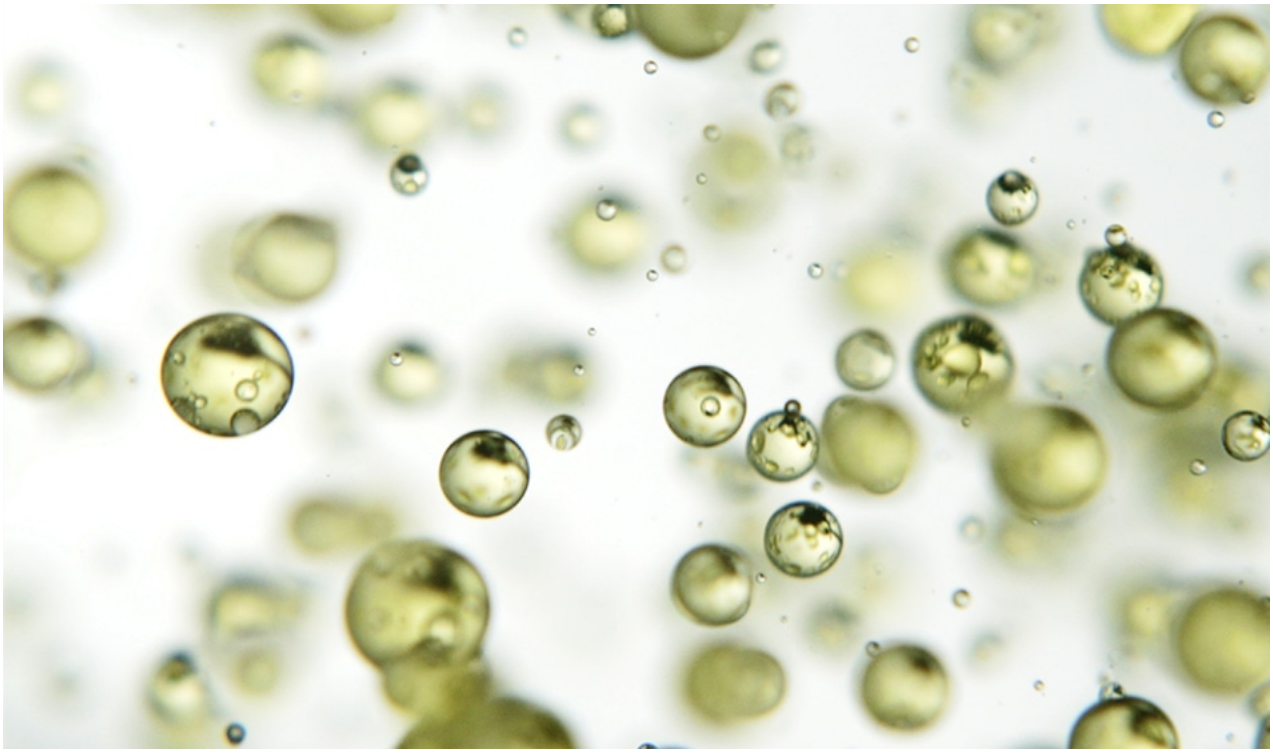
**Benedette Cuffari**

After completing her Bachelor of Science in Toxicology with two minors in Spanish and Chemistry in 2016, Benedette continued her studies to complete her Master of Science in Toxicology in May of 2018. During graduate school, Benedette investigated the dermatotoxicity of mechlorethamine and bendamustine, which are two nitrogen mustard alkylating agents that are currently used in anticancer therapy.

# Elemental Analysis with Laser-Induced Breakdown Spectroscopy

*By Marta Kargol, Ph.D.* Feb 6 2019

**Elemental analysis is an analytical technique commonly used to determine the elemental composition of chemical compounds and their mixtures. This analysis answers what percentage of a sample is made up of chemical elements and what these elements are; providing both qualitative and quantitative analysis.**



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## Why LIBS for elemental analysis?

Laser-Induced Breakdown Spectroscopy (LIBS) is an optical emission spectroscopy technique used to measure elemental concentrations in a material; starting from the complex analysis of ultra-low elemental concentrations in alloys, ores, brines and powders, to basic material or scrap samples.

LIBS is one of the few analytical techniques with the ability to measure a large number of chemical elements simultaneously. This technique can provide multi-elemental analysis, determining the atomic composition of an unknown sample with only a single

measurement.

In contrast to X-ray fluorescence (XRF) and other methods, LIBS offers light chemical elements characterization. The main advantages of the technique are as follows:

- No or minimal sample preparation
- Applicable for solid, liquid, gaseous samples and slurries
- Allows fast scanning of the sample surface and depth profiling
- Ultra-fast measurement time for a single spot analysis
- Sensitivity to light elements (e.g., H, C, N, O, Na, Li)
- Enables thin sample analysis with no substrate interference
- Is mostly a non-destructive technique

Laser-Induced Breakdown Spectroscopy is a competitive technology alongside other commonly used methods for elemental analysis, i.e., Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) and Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES).

## How does LIBS work?

LIBS utilizes a short laser pulse to create a hot plasma on the sample surface for fast and precise chemical analysis. More specifically, the laser ablation process takes place when a small volume of sample mass is removed under a laser beam focused on the surface of the material.

The ablated material consists of free electrons, neutral particles, and ionic species, and expands as a high-temperature plasma. Hot plasma, with a temperature above 10,000 K, possesses enough energy to induce excitation of electrons in the outer orbitals.

Next, the plasma cooling process occurs, and the electrons of the atoms and ions return to a stable state. Finally, the plasma emits light with separate spectral peaks, which are collected and combined with an ICCD detector for further spectral analysis.

Chemical composition can be easily evaluated by identifying the different peaks for the analyzed samples. Each element in the periodic table is linked with unique LIBS spectral peaks. Moreover, the spectral peak intensities enable to quantify the amount of trace and major elements in the tested sample.

The obtained LIBS spectra are characterized by using atomic databases and qualitatively assigned to the adequate elements. On the other hand, quantitative analysis is based on

a calibration method, with reference materials comprising different analyte concentrations. Moreover, depending on the analyte-matrix, low ppm-range of element concentrations can be measured.

## Applications and elemental analysis development

The LIBS technique is suitable for a broad range of samples; from metals, semiconductors, glasses, insulators, plastics, and biological tissues, to coatings and electronic materials. Minerals, natural soils, contaminated soils, rocks, food products, atmospheric particles, and biological materials are often intensively examined using LIBS technology.

Some examples of LIBS technology applications include:

- Elemental analysis of hazardous materials, such as radioactive, high-temperature or toxic materials
- In-situ detection and analysis of radioactive impurities present on storage containers
- In-situ elemental analysis of steel materials in restricted access site environments, e.g., nuclear reactor pressure vessels
- Analytical study of cultural heritage metallic material
- Process control via on-line elemental analysis of liquid metals and alloys or liquid glasses
- In-situ materials analysis immersed in water
- Polymers identifications in waste electrical and electronic equipment (WEEE)

Elemental analysis with LIBS spectra can be divided into *multivariate analysis* (where several peaks of the same element or the entire LIBS spectrum is used) and *single analysis* (where only one peak is used). Multivariate analysis is usually performed using Partial Least Squares Regression (PLSR), which results in better precision and accuracy when compared to one-peak analysis.

Since LIBS is simultaneously sensitive to all elements, a single laser pulse can be used to record the broadband emission spectra, and thus a ‘chemical fingerprint’ of the material can be provided. That's why LIBS is currently being extensively investigated and developed for use outside the laboratory.

Recently, LIBS technology has been miniaturized into a handheld device form - HH LIBS that is capable of analyzing any element, depending on the spectrometer range selected for the device.

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# The Impact Raman Spectroscopy Can Have on the Environment

By Benedette Cuffari, M.Sc. Apr 3 2019

**In order to address the wide range of problems affecting our environment, it is crucial for climate scientists to have the tools necessary to accurately detect, measure, and analyze the chemicals that are often the sources of most ecological issues.**

Although numerous microscopy tools have been used for these purposes, they are often limited in their detection capabilities and capacity to distinguish between different chemical classes.

Over the past ten years, Raman spectroscopy has emerged as a highly precise and unique tool capable of overcoming these common limitations of traditional microscopy techniques in order to evaluate some of the most significant substances affecting the health of this planet.


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## Measuring Aerosol Particles

The small size of most aerosol particle contaminants can prevent environmental analysts from accurately identifying individual particles, especially when their diffraction limit is below 350 nanometers.

Atmospheric aerosol particles have the potential to adversely affect the environment through various mechanisms, some of which include the scattering and absorbing of solar radiation, as well as mimicking the actions of cloud condensation and ice nuclei to modify natural precipitation and cloud properties.

To further complicate matters, atmospheric aerosol particles can contain up to thousands of different chemical species from different sources and different atmospheric ages. Therefore, it is imperative for environmentalists to be capable of quantifying these impacts by analyzing the various physicochemical properties of aerosols.

To address these critical concerns, researchers have often turned to Raman spectroscopy to characterize the properties of aerosol particles. Raman spectroscopy has been used to characterize many different types of particles, the specific compounds present within the aerosol particles, hygroscopic properties, phase separations, heterogeneous reactions, ice nucleation, and acidity.

The minimal sample preparation required for Raman spectroscopy, combined with its non-destructive analysis under ambient temperature and relative humidity (RH) conditions has proven to be especially useful for aerosol particle characterization.

## Surface Enhanced Raman Spectroscopy (SERS) and the Environment

Although Raman spectroscopy alone has proven to be useful for the analysis of aerosol particles, it is limited in its ability to distinguish between particles that are smaller than 1 micrometer (μm) in size. Another limitation of this analytical technique is attributed to its inability to determine different chemical species within the particles when present in lower concentrations.

In an effort to improve the limit of detection for low concentration chemical species, researchers have turned to surface enhanced Raman spectroscopy (SERS). SERS enhances weak Raman signals by exciting electrons in metallic substrates in order to create interactions with localized surface plasmon resonances (LSPRs).

To date, SERS has been used for biosensors, chemical warfare agents, and in conjunction with spectroelectrochemistry, as well being used in many other applications. When applied for the analysis of atmospheric aerosol particles, SERS has demonstrated its ability to accurately detect particles as small as 150 nm in size, as well as their chemical composition and mixing state.

## Identifying Microplastics

The widespread pollution of microplastics within our environment has continued to raise concerns on the damage these contaminants can inflict on all living organisms. Raman spectroscopy has demonstrated its ability to accurately detect microplastics as small as 20 µm. Furthermore, when Raman microscopy is used in conjunction with advanced detectors and spectrum processing devices, researchers have been able to achieve enhanced signal quality.

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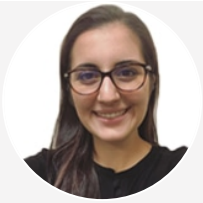

**Written by**

## Benedette Cuffari

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# Can Infrared Spectroscopy Be Used in Mineral Characterization?

By Brett Smith, B.A. Apr 11 2019

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**Infrared spectroscopy is a well-established technique for mineralogy investigation that requires little or no sample preparation. IR spectroscopy is often used when other techniques take too long or require the destruction of the sample. When it comes to the characterization of minerals in a sample, IR spectroscopy can provide a comprehensive overview.**

IR mineralogical techniques can be applied to crystalline, non-crystalline, organic and inorganic materials. This approach allows for the qualitative investigation of a large range of specimens ranging from biological to clay minerals. Identification of unknowns is achievable through expert interpretation of spectra in conjunction with the usage of spectral libraries. Even though IR evaluation is mostly qualitative, quantitative work can also be done. Near-infrared spectroscopy (NIR) is a related process that allows for the quantification of various mineral characteristics in a wide range of materials, from food products to soil.

The IR spectrum generated by a mineral evaluation is the chemical fingerprint of the test sample. It is normally plotted as absorption against frequency. Translation of IR spectra is accomplished by using functional-group frequency tables, spectral database searches, and identification of absorption patterns based on experience. IR spectroscopy can be very helpful in the identification and description unknown specimens but requires special care to ensure spectral libraries are used correctly, particularly where spectra are derived from complicated mixtures.

Close evaluation of the spectrum from a sample is based on database spectra, but a strong match between test results and a library spectrum does not always mean a positive identification. All the bands from the sample ought to match the reference spectrum, not just a couple, for positive detection.

A Fourier transform infrared (FTIR) spectrometer is often used in a laboratory mineral analysis. Analysts may use a Diamond Attenuated Total Reflectance (DATR) holding accessory to enable specimens to be tested in their normal state and be recovered afterward. A Horizontal Attenuated Total Reflectance (HATR) accessory allows for the FTIR spectrometer to test mineral liquids and solutions.

FTIR microscopes have been used for everything from the identification of fibers to determination of chemical composition in biological thin films.

## Use in Space-Based Research

Space-based spectrometers are becoming increasingly sensitive and researchers are using them to acquire precise mineral identification and mineral quantity values from mixed spectra. The spectra generated by these IR spectrometers are also compared to spectral libraries, which were produced under modeled conditions in terrestrial laboratories.

Comprehensive reports of mineral structures are most often conducted under ambient temperatures as it is experimentally convenient, especially far from Earth. However, temperature-dependent issues can considerably alter the behavior of various mineral species in a crystal lattice.

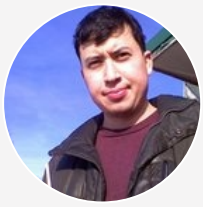
IR spectroscopy is often utilized due to researchers' current focus on hydrated minerals and IR’s level of sensitivity to the O-H bond, in addition to the relative simplicity of acquiring experimental data at temperatures both colder and warmer than ambient conditions.

IR spectroscopy is highly responsive to both mineral composition and species concentration. The latest developments in remote sensing and communications have heightened the usage of IR spectroscopy in non-ambient conditions. As a result, the low-temperature responses of hydroxyl (OH-) and molecular water (H2O) species have become essential to the search for hydrated minerals on extra-terrestrial surfaces. Researchers are particularly interested in the hydrated mineral inventory on asteroids because they can function as a tracer for major events taking place during the development of the solar system, and the moderate-temperature response of hydrated minerals is also relevant in the study of water traveling to the Earth's interior from subduction zones.

## Resources

- https://hayesresearchgroup.com/research/mineral-spectroscopy/
- https://www.claysandminerals.com/methods/infraredspectroscopy
- https://speclab.cr.usgs.gov/PAPERS.refl-mrs/refl4.html#section9.0





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APPLICATION NOTE



# Total oxide X-ray analysis with ARL 9900 IntelliPower Series Simultaneous/Sequential X-ray Fluorescence Spectrometers

**Author:** Didier Bonvin
Product Manager, XRF

## Introduction

Wavelength-dispersive X-ray fluorescence (WD-XRF) allows measurement of up to 83 elements of the periodic table in samples of various forms and nature: solids or liquids, conductive or non-conductive. Advantages of XRF over other techniques are speed of analysis, generally easy sample preparation, very good stability, precision and wide dynamic range (from ppm levels to 100 %).

Accuracy of analysis of powders can be impaired by particle size effects and mineralogical effects. Although inhomogeneities and particle size effects can often be minimized by grinding below 50 microns and pelletizing at high pressure, often mineralogical effects cannot be completely removed or harder particles cannot be broken down below the required size.

**Figure 1:** Many different materials can be analyzed with an ARL 9900 spectrometer calibrated with our General Oxide calibration.

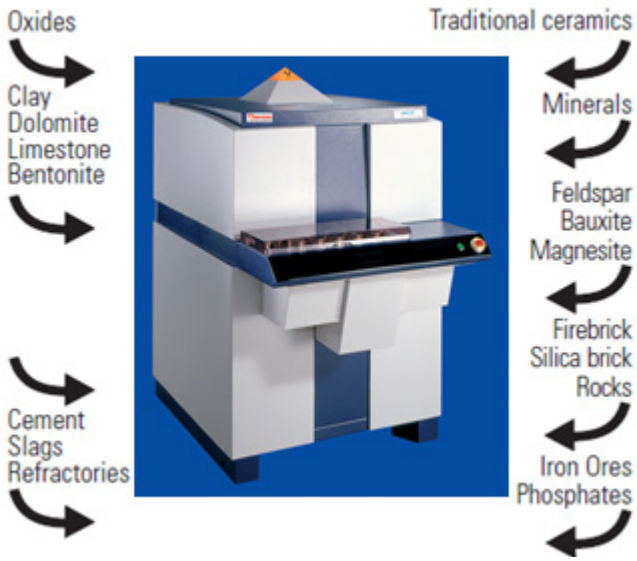
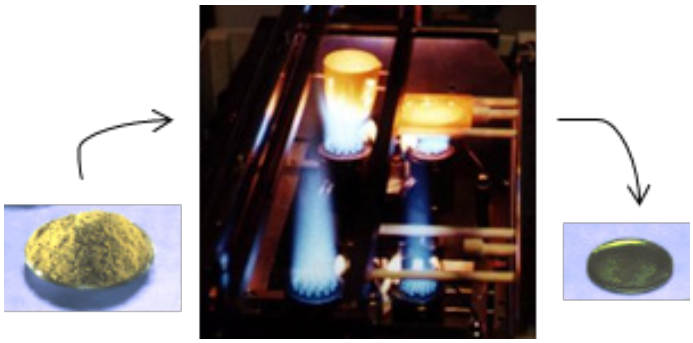



**Figure 2:** Transformation of the powder material into a glassy sample by fusion at high temperature.



Fusing these oxidic materials is the best way of completely removing both grain size and mineralogical effects. Essentially, the procedure consists of heating a mixture of sample and a borate flux, namely lithium tetraborate and/or lithium metaborate at high temperature (1000°-1200°) so that the flux melts and dissolves the sample. The overall composition and cooling conditions must be such that the end product after cooling is a one phase glass.

The Thermo Scientific™ ARL™ 9900 Series X-ray Fluorescence Spectrometer can be calibrated from the factory as a complete analytical package which provides the possibility to analyze a large variety of minerals (Figure 1), using the general oxide calibration based on a sample preparation by fusion (Figure 2).

## Calibration ranges and results

The types of oxides that are addressed and their concentration ranges are shown in Table 1. A working curve is established for each element using the multi-variable-regression incorporated in the "state-of-the-art" Thermo Scientific™ OXSAS™ Software package. Theoretical alpha factors are used for all matrix corrections. Loss on ignition values, which spread up to 47% can be used for correction purposes in the multi-variable regression. The standard error of estimate (SEE) is a measure of the accuracy of analysis. It is the average error between the certified concentrations of the standard samples and the calibration curve for a given oxide.

The limits of detection (LOD) determined with precision tests at low concentrations are listed in Table 2 for the various oxides when the universal goniometer is used. The analysis time per element can range from 4 to 40 seconds depending on the element and the precision required. Obviously the total counting time can be decreased substantially when fixed channels monochromators are used for several elements/oxides.

**Table 1:** Concentration ranges of the various oxide types with the Standard Errors of Estimate (SEE) achieved.

| Elements | Range [%] ignited samples | SEE [%] |
|---|---|---|
| $Al_2O_3$ | 0.04 - 89.2 | 0.16 |
| CaO | 0.006 - 94.4 | 0.32 |
| $Cr_2O_3$ | 0.05 - 17.2 | 0.03 |
| $Fe_2O_3$ | 0.05 - 93.9 | 0.15 |
| $K_2O$ | 0.06 - 15.4 | 0.03 |
| MgO | 0.03 - 96.7 | 0.22 |
| MnO | 0.01 - 5.2 | 0.03 |
| $Na_2O$ | 0.03 - 10.06 | 0.05 |
| $P_2O_5$ | 0.01 - 37.83 | 0.15 |
| $SO_3$ | 0.015 - 2.95 | 0.05 |
| $SiO_2$ | 0.26 - 99.9 | 0.23 |
| $TiO_2$ | 0.01 - 3.8 | 0.03 |

## Sample preparation

Standard samples are dried prior to being fused as shown in Figure 2. Standards are prepared from ignited or non-ignited powder as 35 mm diameter fused beads. Ignition is carried out for one hour at 1050°C when required. The fusion is made from 0.7 grams of sample, 7.7 grams of Fluorex 65 and 0.02 grams of LiBr (dilution 1:11) on a Katanax electrical fusion machine or a gas fusion machine (Vulcan or Fluxana).

Two types of sample preparation can be used:

**a. No calcination of samples**

*Quicker preparation for clean oxides*

Loss on ignition is estimated by the software, therefore all elements must be measured for this automatic correction to work. If other elements/oxides than the 12 measured are present, the loss on ignition should be introduced through manual input in order to improve accuracy of analysis. Note that fusion from non-ignited samples can be fatal to the Pt-Au crucible in case small metallic particles are present in the sample.

**b. Fusion from ignited samples**

*Better accuracy and safer fusion*

Samples are ignited at 1050°C for 1 hour and their loss on ignition (LOI) is determined. Samples are prepared from ignited powder as 35 mm diameter fused beads. Ignited samples are easier and safer to fuse especially in case small metallic particles are present. Samples prepared by both methods can be analyzed using the same calibration curves.

**Table 2:** Typical limits of detection on ARL 9900 Intellipower Series in 100s obtained on various oxides (fusions with 1:12 dilution).

| | 3600 W (3σ) [ppm] | 2500 W (3σ) [ppm] | 1200 W (3σ) [ppm] |
|---|---|---|---|
| CaO | 12 | 14 | 20 |
| $SiO_2$ | 13 | 15 | 22 |
| $Al_2O_3$ | 32 | 38 | 55 |
| $Fe_2O_3$ | 12 | 14 | 20 |
| MgO | 74 | 89 | 128 |
| $Na_2O$ | 143 | 172 | 248 |
| $SO_3$ | 17 | 20 | 29 |
| $K_2O$ | 10 | 12 | 17 |
| $P_2O_5$ | 17 | 20 | 29 |
| MnO | 8 | 9 | 13 |
| $Cr_2O_3$ | 7 | 8 | 12 |
| $TiO_2$ | 7 | 8 | 12 |

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## Factory pre-calibration

The pre-calibration of the ARL 9900 WDXRF Spectrometer can be carried out directly in the factory prior to delivering the spectrometer to the client. We use certified standard samples prepared on a Katanax electrical fusion machine or a gas fusion machine (Vulcan VAA2 or Fluxana depending on customer choice). No standard samples are delivered with this pre-calibration, but a set of six stable and polished setting-up samples for maintenance of the calibration curves over time.

Alternatively a kit of 24 international certified standards of oxide materials is available to allow the customer to calibrate the instrument on-site using his own sample preparation equipment.

## Conclusion

These results show that various types of minerals, raw materials as well as oxidic products can be analyzed with good accuracy and precision by coupling wavelength dispersive X-ray fluorescence and a sample preparation as fusion beads.

Thanks to a clever management of power, the ARL 9900 Intellipower Spectrometers can operate up to 2500 W without requiring external water cooling. Therefore neither tap water, nor a water cooler is required in these cases. At higher power levels (3.6 kW or 4.2 kW), energy savings and reduced stress on the X-ray tube are obtained thanks to intelligent management of the X-ray tube power. The configuration of the ARL 9900 Intellipower Spectrometers can be chosen either with just one sequential goniometer or with the addition of fixed channels to speed up the response time.

Furthermore the state-of-the-art OXSAS Analytical Software under Windows® 10 provides comprehensive analytical functions and ease of use.

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