

University of Bath

An Investigation into the Spectroscopy Techniques used for Analysis of Lunar Mission One's Borehole Findings

Nuffield Research Report 2016

Philippa Darling-Holmes

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INTRODUCTION

This project is set out to fully understand why the chosen analytical chemical techniques are going to be used, and how they will work whilst on the moon. On the subject of analytical chemistry (and by extent astrochemistry), I knew very little about spectroscopy in space, my only prior knowledge being about the use of a Miniature Thermal Emission Spectrometer (Mini-TES) used on the Mars Rover to monitor infra-red radiation emitted from the soil and rocks on the surface of Mars. The issue here is that this is not specifically about our moon, so I needed to do a lot more research into previous spectrometer methods used on the moon, and gain a full understanding about the specific spectrometry methods that the Lunar Mission One will be using. I found my information looking in textbooks and general interest books that I located in The University of Bath's Library, and looking online, using search engines tailored for academic research such as 'Virtual LRC' and 'iSeek Education'.

From the Lunar Mission One project website, through reading their online Technical Review, an understanding for what the Mission was aiming to achieve via their cosmo-chemical experiments that the unmanned spacecraft will carry out whilst on the moon, can be gauged. The review roughly outlines the Mission's plans to for the lunar surface. In brief these include dating the South Pole Aitken basin (the largest impact crater in the solar system, roughly 2500km wide and 13 km deep) ^{[1],} a clearer understanding of the moon's geochemistry and mineralogy. There are also plans for a volatile examination, thermal profiling (a complex set of time-temperature data), radio astronomy and measuring the radiation, dust and charging environment. ^[2] This is alongside the plans to assess the south pole's suitability for a future permanently manned lunar base, and the placement of a 'Life on Earth' archive inside the borehole, that will be preserved for millions of years to come. ^[3] The review also indicates plans to use a range of spectroscopy methods, including Raman LIBS, gammaray, Neutron and Mass spectrometry. This report will be looking into why these methods were chosen, and how they will be used in a range of experiments to give us results for Lunar Mission One's plans for the South Pole Aitken Basin.

ABSTRACT

Chemical analysis has occurred on the moon for just over 50 years, using a range of spectroscopic techniques, but nobody, and no space craft, has ever dug lower than a few centimetres below the moon's surface. This report is an investigation into the use of a range of analysis techniques that will be used on this unique mission to dig nearly 100 meters below the moons' surface, and to see what we will begin to understand about the moon and its history by doing so.

METHODOLOGY

To carry out this report, the primary form of research was done using academic search engines such as Google Scholar, Research Gate, iSeek Education, and Web of Science, to read and learn about relevant content from published academic journals. The first steps were to locate the appropriate keywords to use as this form of investigation was carried out, by reading a range of textbooks and other non-fiction literary volumes from University and public libraries. The best way to start my online research was to begin at popular and relevant sites such as NASA and the Lunar and Planetary Institute. By navigating through fitting hyperlinks and content-specific journals, the research project was able to be developed to a great breadth and depth.

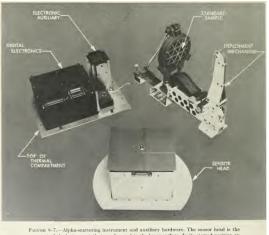
RESULTS OF RESEARCH

The History of Chemical Analysis on the Moon

The earliest examples of chemical analysis being used on the moon can be seen in the Surveyor Missions in 1967 and 1968. Set up and carried out by NASA, by the work of Urey, they too were unmanned, and were initially intended to be a series of robotic scientific missions, but the focus of the program was changed to engineering to support the preparations of the manned Apollo moon landing that happened around a year later on 20th of July 1969. Alongside testing landing techniques designed for a safe landing for the astronauts, the Surveyor Missions (of which five of the seven successfully landed), took close-up images of the lunar surface, and missions 5, 6 and 7 used an alpha-backscatter instrument to help pinpoint appropriate landing locations. Rutherford Backscattering Spectrometry determines the structure and composition of materials by measuring

the backscattering of a beam of high-energy ions impinging on a sample.^[4] The Surveyor Mission's Alpha-backscatter instrument used alpha particles as their high energy ions (their 'projectile'). Backscattering spectrometry works by measuring the energies of backscattered ions during an elastic collision between the kinetic projectile and a stationary particle in a sample (a target).

In the Surveyor Missions 5, 6 and 7, the sample was lunar soil, collected in a sample only a few atoms thick, which was collected by the deployment mechanism (pictured). The mechanism consisted of a platform that held the sensor head and a device that could lower the sensor head to the lunar surface on command from Earth. This sensor head is where the chemical analysis took place. The technology in the sensor head consisted of six source capsules that provided the alpha particles, two detectors to register the number and energy of the scattered alpha particles, and four detector systems for the protons. ^[5]The data collected from these tests gave NASA



part of the instrument that was lowered to the limar surface. In its stowed position on the spaceraft, the same head was held on the deployment mechanism on top of the standard sample. The digital electronic and electronic auxiliary were contained in elec-

This diagram shows the Surveyor 5 deployment mechanism for the Alphabackscattering instrument. Source: NASA (1969), *Surveyor Program Results*.

the abundances of the major elements of the lunar surface, telling NASA that the surface of the moon was powdery dust, but strong enough to support the weight of people and machines. The moon had no global magnetic field or atmosphere and was made up of common rock types, similar to those found on Earth. This allowed for the first plans of a manned mission to be put into action, leading to the Moon Landing of 1969. ^[6]

A more recent and relevant project that took place, also operated by NASA was the Lunar Prospector Mission that took place from 7th of January 1998 through to 31st of July 1999, the difference here being that this was a spacecraft that didn't actually land, it instead orbited the moon. It conducted chemical experiments about the moon's surface for a variety of reasons, including aiding scientists about the origins of the lunar landscape, mapping the surface composition, and locating lunar resources, measuring magnetic and gravity fields, and studying outgassing events. Outgassing is where volatiles are vented from deep in the lunar interior to the surface, specifically, radon, nitrogen, carbon monoxide, and carbon dioxide, may be the result of low level volcanic or tectonic events on the moon. ^[7] This 570-day project used spectrometry systems that are relevant to the work that will be conducted in Lunar Mission One, these being a Gamma-ray Spectrometer and a Neutron Spectrometer. Much like the Surveyor Missions 5, 6, and 7, the Lunar Prospector Mission used an Alpha Particle Spectrometer, an instrument that will not be present in the Lunar Mission

One Spectrometer Suite. Their Gamma Ray Spectrometer weighed 8.6kg, and used 3 watts of power, and worked by detecting gamma rays that came from two sources, the first and more prominent here 'Natural' gamma rays (that are emitted spontaneously by the radioactive elements on the moon's surface). The second type of gamma ray ('Induced' gamma rays), that are emitted by more stable elements such as iron, oxygen and silicon when they are effectively 'hit' by cosmic rays. ^[8] A cosmic ray is a highly energetic atomic nucleus or other particle travelling through space at a speed approaching that of light. ^[9]

The history of both manned and unmanned missions that that have involved or have led to chemical analysis of the moon's chemical composition mean that we already know a lot about the regolith. The regolith is the layer of solid material covering the bedrock of the moon. Samples brought back from Apollo 11 were made of Basalt (A dark and porous rock formed by cooling lava). The chemistry of the rocks showed abundant signs of refractories (elements with a high boiling point), and showed that 98% of the crystalline material on the surface of the moon is made up of only 4 elements: Plagioclase Feldspar (CaAlSi₂O₈, calcium aluminium silicate); Olivine ((Mg, Fe)₂SiO₄, magnesium-iron silicate); Pyroxene (FeSiO₃, iron silicate); and Ilmenite (FeTiO₃, iron titanium oxide). These elements, when compared to the Earth, show that the moon has a lower concentration of volatile elements, suggesting a high temperature origin. ^[1]

What Information will Chemical Analysis give about the Moon, and furthermore, the Earth?

Chemical analysis can tell us the chemical construction of any molecule if you know what to look for, and on the moon this is the same case. What Lunar Mission 1 is looking out for is the geological composition of the rock beneath the lunar surface to help identify its origins, if what is dig up has a chemical make-up similar to Earth's, then this is further evidence to support the idea that the moon came from the fragments of Earth, an idea known as the 'giant impact hypothesis' by Hartman and Davis. It states that a planet sized rock, nicknamed Theia, collided with Earth, and that its impact caused part of Earth's outer layer to spin off and form a giant molten ball, which cooled and moved further away over time, that we now know as the Moon. A very large and pressing issue with this theory at present is that from the lunar rocks that have been collected from previous NASA missions, the isotopes present in samples should have origins that are a mix of Earth and from Theia, so the isotopic composition should be somewhere between the two. This is not the case, the lunar rock collected only shows an isotopic make-up that is almost exactly the same as Earth's. The abundance of 'siderophiles' (elements that are 'iron-loving', and have a tendency to alloy with iron) is almost exactly the same, and the oxygen isotope ratios of for moon rocks are indistinguishable from Earth's (showing a large concentration of oxygen-17 and oxygen-18) relative to the stable isotope (oxygen-16). If Theia existed, then it has left no trace on the Moon. If the results of Lunar Mission One's analysis of the borehole rock finds differences between the Earth and moon's geological composition, then we would have the first account of valid evidence that the giant impact hypothesis is the most accurate theory for the moon's creation. On the other hand, the evidence Lunar Mission One will collect could entirely disprove this hypothesis, making way for alternatives that could finally bring about the most valid theory.

Lunar Mission One also intends to date the South Pole Aitken base, the largest known crater in the solar system, and the landing location for the Lunar Mission One's spacecraft, by analysing the rock we find. If we use radiometric dating and the principles of determining relative age of rocks, we can date the crater effectively. This means that by using chemical analysis, we can determine the relative proportions of radioactive isotopes present in a sample. All rocks contain small amounts of unstable isotopes and the daughter isotopes into which they decay. Where the amounts of parent and daughter isotopes can be accurately measured, the ratio can be used to determine how old the rock

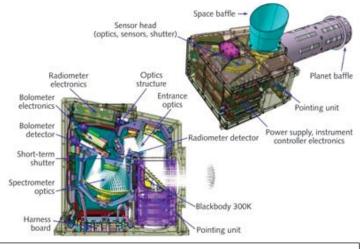
is. We would expect the age of the crater to be between 3.8 and 4 billion years old, based on the 1974 lab work that found out that the other major basins are all this age too. If this is the case, it gives rise to the validity of the late heavy bombardment theory by G. J. Wasserburg, which states that a violent spasm of meteorites bombarded the inner solar system around that time which would have been the cause of this crater.

If we can fully validate and accept the late heavy bombardment period, then this would set a time for when the first signs of life appeared on Earth. This is because nothing could have survived the impacts seen on the moon, such as the South Pole Aitken basin. Earth would have had bombardment of a magnitude much greater than the moon's, due to its surface area being 92.6% bigger than the moon (making it a larger target), alongside a larger gravitational pull on the meteorites due to its increased mass. When Earth cooled down from this intense period, primitive life forms seem to have appeared around a reasonable time afterwards, which must have been a thermophilic strain of bacteria, as these are the first organisms that could have survived such conditions. ^[1]

How will the Chemical Analysis Operate?

Infra-red Spectrometry

Infra-red spectrometry is historically the most common strand of spectrometry used for chemical analysis on the surface of planets, moons, and asteroids. Lunar Mission One's space module will be using an Infra-red Imaging Spectrometer to characterize the local mineralogy (identify functional groups, confirm the structure of compounds, and measure concentrations of particular species).^[2] A recent use of infra-red spectroscopy in space is the Mars Rover mission, which used a miniature thermal emission spectrometer (Mini-TES) to measure the infrared radiation emitted from the soil and rocks on the surface of mars as a result of the vibrations of chemical bonds. The use of an Infra-Red imaging spectrometer is relatively uncommon, it's only use on the past being on Earth, to



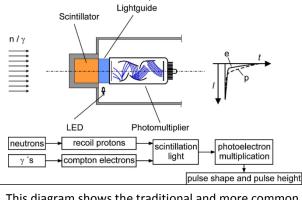
This image shows the internal instruments of infrared imaging spectrometry, and is labelled accordingly Source: http://www.laserfocusworld.com/content/dam/lfw/printarticles/2012/07/1207LFW03f3

measure similar things to what Lunar Mission One will learn about the moon from this technique, but also vegetation and ecology detection. This system worked in by traveling the circumference of the earth whilst in orbit, so unlike Lunar Mission One, there was and still is no intention to use it on the Earth's surface (hence it's name- ARVIS, meaning 'Airborne Visible/Infra-red Imaging Spectrometer).

The primary function of an Infra-red imaging spectrometer is to use hyperspectral imaging. 'Hyperspectral imaging' collects and processes information from across the electromagnetic spectrum, the aim being to obtain the spectrum for each pixel in the image of a scene, with the purpose of finding objects, identifying materials, or detecting processes. These are all essential to carry out Lunar Mission One's plans for a characterisation of local mineralogy, and to measure concentrations of species. Imaging in the emissive region of the electromagnetic spectrum (waves that have the power to emit something such as light or heat) typically involves the section of the electromagnetic spectrum associated with primarily vibrational motion of molecules. A common analogy for the vibration of a chemical bond is a diatomic molecule acting like they are connect by a spring, and are able to stretch, compress and bend a little. The analysis of this motion occurs as the vibrational energy of a bond is quantized (they are restricted to discrete values instead of continuous values). These molecular vibrations occur in the mid infra-red and the long wave infra-red, meaning their covers the range of the electromagnetic spectrum between 0.78 and 1000 mm. For a molecule to absorb Infra-red, the vibrations within a molecule must cause a net change in the dipole moment of the molecule. The alternating electrical field of the radiation (considering that electromagnetic radiation consists of an oscillating electrical field and an oscillating magnetic field, perpendicular to each other) interacts with fluctuations in the dipole moment of the molecule. If the frequency of the radiation matches the vibrational frequency of the molecule then radiation will be absorbed, causing a change in the amplitude of molecular vibration. Analysis of the hyperspectral data across this portion of the spectrum deal with the reflective nature of solid and liquid materials (the minerals that are being analysed on the moon). This collection of different transitions, leading to different radiated wavelengths, make up an infrared spectrum. Each element's emission spectrum is unique. Therefore, spectroscopy can be used to identify the elements in matter of unknown composition.

Neutron Spectrometry

Neutron Spectrometry will be used to determine the concentrations of hydrogen in the regolith. The commonly available techniques for the determination of hydrogen dissolved in solids are usually destructive from the point of view of the sample, as seen on the Messenger probe on Mercury in 2011. ^[12] A new, non-destructive method for these kinds of measurements has been developed,



This diagram shows the traditional and more common Neutron Spectrometer Source:http://whyfiles.org/060moons/ourmoon2.html

requiring less energy than previous neutron spectrometers, with the requirement of improved sensitivity for massive samples. This scattering method is based on the use of extremely hot neutrons, and has been implemented through the design and construction of a spectrometer dedicated to that task. ^[11] The diagram shows a traditional version of the neutron spectrometer, as the newer design is still in progress. On similarity though, is that the traditional transmission method has been employed to determine hydrogen content in minerals, using the full sub-themal and thermal

neutron energy ranges. A pulsed neutron source (based on an electron linear particle accelerator) is

employed, together with time-of-flight techniques. An electron linear particle accelerator is a type of particle accelerator that greatly increases the kinetic energy of charged subatomic particles (electrons) by subjecting the charged particles to a series of oscillating electric potentials along a linear beam line. ^[13] If Lunar Mission One's Neutron Spectrometer finds evidence of hydrogen lower down in the moon, it will further postulate that the Earth and the moon are made of very similar materials, giving further evidence to support the giant impact hypothesis.

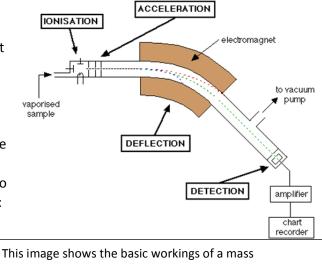
Neutrons are electrically neutral sub atomic particles that, together with positively charged protons, make up the centres or nuclei of atoms, the spectroscopy technique measuring the atomic and magnetic motions of atoms. This strand of spectrometry uses inelastic neutron scattering, which measures the change in the energy of the neutron as it scatters from a sample. This can be used to probe a wide variety of different physical phenomenon: diffusional or hopping motions of atoms, the rotational modes of molecules, sound modes and molecular vibrations, recoil in quantum fluids,

magnetic and quantum excitations or even electronic transitions. The vibrational motion of atoms is, entirely or in part, responsible for a large number of the characteristic properties of a material, such as the specific heat, thermal conductivity, optical and dielectric properties and electrical resistance, but it is also a direct way of understanding the nature of atomic bonding. The most commonly used spectroscopies are the light scattering techniques: Raman and infrared, but are only sensitive to certain vibrational modes. Inelastic neutron scattering is usually used to understand the physics of a system. It is a highly quantitative probe whose results are directly comparable to numerical and analytical calculation. It can be used to understand the nature of a phase transitions or linked directly to thermodynamics quantities, like specific heat or thermal conductivity, or structural properties such as force tensors or bulk and shear moduli. Due to the unique nature in way that hydrogen in a material. Hydrogen is one of the few elements that has a negative scatter, which means that neutrons deflected from hydrogen are 180° out of phase relative to those deflected by the other elements.

Mass Spectrometry

Mass Spectrometry is required to analyse the chemical composition of the sampled materials when vaporised. Mass Spectrometry works as follows: Atoms and molecules are deflected by magnetic fields - provided the atom or molecule is first turned into an ion. This will be done to the sample upon entry into the system (labelled 'ionisation' on the diagram). Electrically charged particles are affected by a magnetic field although electrically neutral ones aren't. ^[14]

The four key stages of a mass spectrometry sequence are ionisation, acceleration, deflection, and detection. The sample from the borehole will be ionised on the spacecraft by collision of the gaseous molecules at low pressures when an electric current is passed through them. Another option is intermolecular collisions at high temperatures, but this is less effective. This may cause some of the sample's molecules to break into charged fragments. These ions are then separated according to their mass-to-charge ratio, typically by accelerating them and subjecting them to an electric or magnetic field, created by an electromagnet: ions of the same mass-to-charge ratio will undergo the same amount of deflection; lighter ions will undergo a deflection of greater magnitude compared to heavier ions. ^[15] The detection of the ions is done by sorting the ions based on their mass to charge ratio. A mass spectrum is then compiled, and it is a plot of the ion



spectrometer. Source:http://www.chemguide.co.uk/analysis/masspec/ howitworks.html

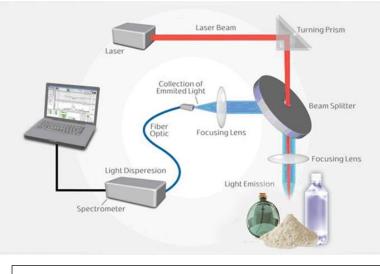
signal as a function of the mass-to-charge ratio. These spectra are used to determine the elemental or isotopic signature of a sample, the masses of particles and of molecules, and to elucidate the chemical structures of molecules. The atoms or molecules in the sample can be identified by correlating known masses to the identified masses or through a characteristic fragmentation pattern. ^[16]

The equipment require to carry out mass spectrometry is relatively simplistic compared to other spectrometry techniques. The first stage (ionisation) occurs using an ion source, which consists of an ionising filament (an electrically heated metal coil), an ion repeller, and an electron trap. The ionising filament releases electrons which are attracted to the electron trap which is a positively charged

plate. The particles in the sample (atoms or molecules) are therefore bombarded with a stream of electrons, and some of the collisions are energetic enough to knock one or more electrons out of the sample particles to make positive ions. Acceleration occurs by repelling the positive ions away from the very positive ionisation chamber and pass through three slits, the first being at 10000 volts, and the final one of which is at 0 volts. The middle slit carries some intermediate voltage. All the ions are accelerated into a finely focused beam. Deflection is done using an electromagnet, the electricity coming from Lunar Mission One's solar panels on the outside of the space craft. Detection is commonly done using an electron multiplier, which is a vacuum-tube structure that multiplies incident charges. An incident charge being the charge of a deflected ion coming into or entering the surface of the equipment. If an electric potential is applied between the two metal plates on the electron multiplier, the emitted electrons will accelerate to the next metal plate and induce secondary emission of still more electrons. ^[17] This can be repeated a number of times, resulting in a large shower of electrons all collected by a metal anode, all having been triggered by just one.

Raman-LIBS

Raman LIBS, meaning 'laser induced breakdown spectrometry', is necessary for this mission to determine the elemental composition of the borehole findings, as well as the mineralogy. Raman spectroscopy works by providing information about molecular vibrations by shining a monochromatic light source on a sample and detecting the scattered light. ^[18] This will give the Lunar



This image shows the innate workings of a standard Raman-LIBS Source: http://laser-detect.com/technology-methods/

Mission One team information about sample identification and guantitation relevant to the mineral content of the borehole rock. The majority of what is detected is known as Rayleigh or 'elastic' scattered light, which has the same frequency as the excitation source. The minority of what is detected (about 0.0001%) is the shifted light, the scattered light that has shifted in energy from the laser frequency due to interactions with the sample's elements. The results are formed by plotting the intensity of the shifted light against the frequency, and the band positions correspond to energy levels of different functional group vibrations, allowing for identification. ^[19]

There doesn't appear to be any examples of Raman spectrometry used in space, but on earth it gives very accurate results, compared to other spectroscopic techniques such as Neutron Spectrometry, Raman LIBS is cheap, readily available, and highly sensitive.

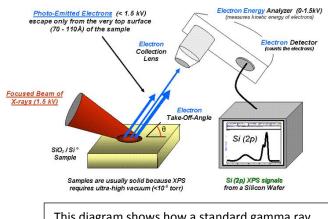
The equipment used in Raman LIBS will have to be tailored to what the mission is looking for, in terms of the mineral, Lunar Mission One knows to expect the larger molecules that make up the crystalline material mentioned earlier (see 'History of Chemical Analysis on the Moon) so will need to use longer wavelength light (such as green or red, but red is more common) to allow for significant scattering. The stereotypical Raman LIBS has equipment shown in the image, the turning prism being a hypotenuse-coated right angle polyester or glass prism coated in a usually metallic substance to reflect the light towards the beam splitter. In Raman LIBS, the beam splitter is most commonly a cube, made from two triangular prisms glued together. The thickness the resin layer is

adjusted such that (for a certain wavelength) half of the light incident through one "port" (i.e., face of the cube) is reflected and the other half is transmitted due to frustrated total internal reflection. [20] 'Frustrated total internal reflection' is a phenomenon which occurs when a propagating (leaving or spreading) wave strikes a medium boundary at an angle larger than a particular critical angle with respect to the normal to the surface. This allows the laser beam to be split and focussed, to then be transmitted down the optical fibre to carry out the spectrometry in the spectrometer. This is where all the detection and Rayleigh scattering takes place, and then finally the data is collected by a computer, and converted into a spectrum to allow for data analysis.

Gamma Ray Spectroscopy

Gamma Spectroscopy is required on this mission to determine elemental composition, mineralogy,

trace elements and radio-nuclides from within the moon by analysing the borehole rock. The overall performance of the spectroscopic method has proven to be quite successful in previous investigations on the moon's surface. The 1998 Lunar Prospector mission used a gamma ray spectrometer to measure the abundances of 10 different elements such as uranium, iron, oxygen and silicon. The results here showed signs of natural gamma radiation, and induced gamma radiation as a result of cosmic rays. Gamma ray Spectroscopy is a quantitative study of the energy spectra, which is the intensity of a particle beam as a function of particle energy of gamma ray sources. Gamma-Ray spectroscopy detectors are passive materials that wait for a gamma interaction to occur in the detector.



This diagram shows how a standard gamma ray spectrometer works. Source: http://inspirehep.net/record/1086599/plots

Some constructions of scintillation counters can be used as gamma-ray spectrometers. The gamma photon energy is recognized from the intensity of the flash of the scintillator, a number of lowenergy photons produced by the single high-energy one. A scintillation counter is an instrument for detecting and measuring ionizing radiation by using the excitation effect of incident radiation (as seen in the emission infra-red spectrometry in the Mini-TES used on the Mar's Rover) on a scintillator material (a material that fluoresces when hit by a charged particle), and detecting the resultant light pulses. This is more likely to be the preferred kind of Gamma-Ray Spectrometry, as the alternative (using pure germanium that produces pulses proportional to the captured photon energy) requires bulky apparatus. Scintillator kind gamma ray spectrometers are also lighter and easier compressed, which will allow for easier transportation for use on the moon. The scintillator material used is mostly thallium-doped sodium iodide (NaI(TI)), thallium-doped caesium iodide (CsI(TI)), or, more recently, cerium doped lanthanum bromide (LaBr₃Ce).

When exposed to the aforementioned cosmic rays, chemical elements in the soil and rock emit uniquely identifiable signatures of energy in the form of gamma rays. When nuclei are hit with such energy, neutrons are released, which scatter and collide with other nuclei. The nuclei get "excited" in the process, and emit gamma rays to release the extra energy so they can return to their normal rest state. ^[9] Some elements like potassium, uranium, and thorium are naturally radioactive and give off gamma rays as they decay, but all elements can be excited by collisions with cosmic rays to produce gamma rays. The gamma-ray spectrometer looks at these signatures, or energies, coming from the elements present in the target borehole sample. By measuring gamma rays that originated from the target body (the lunar rock), it is possible to calculate the abundance of various elements. Gamma rays, emitted from the nuclei of atoms, show up as sharp emission lines on the instrument's spectrum output that will be transmitted back to Earth from the space module. While the energy represented in these emissions determines which elements are present, the intensity of the spectrum reveals the elements concentrations. ^[22]

CONCLUSION

The history of the use of chemical analysis on the moon is a rich one, and it sets the stage for Lunar Mission One to make bold strokes into the progression of our understanding and possibly alter our knowledge of the origins of the moon as we know it today. The uses of spectrometry methods to carry out this feat are well chosen, and will allow for a broad analysis of the borehole findings in terms of geochemistry, mineralogy, concentrations of hydrogen, a deeper look into elemental composition and volatile presence, alongside the lunar environment.

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