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Review and Extension of Advances in Zero-Field Nuclear Magnetic Resonance Spectroscopy to Monitor Tholin Formation on Titan

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***Abstract*: This paper explores the recent advancements in the field of NMR spectroscopy and proposes adapting this technique to study tholin formation on Saturn's moon Titan. Reviewing the UC Berkeley paper: 'Advances in Zero-Field Nuclear Magnetic Resonance Spectroscopy', which pioneered no-field NMR spectroscopy. This paper details various components and changes made to the technique since its conception, such as the use of PHIP, μ metal, nitrogen buffer gas, pump and probe lasers, which make NMR spectroscopy more accessible for space exploration. In addition to detailing the existing developments, this paper also specifies the key changes required to make NMR spectroscopy a feasible analytical technique for studying the formation of tholins. Our aim in this paper is to observe tholin formation so as to better understand the origin of life in the universe via the help of no-field NMR spectroscopy. The paper will include an overview on the principles behind NMR Spectroscopy, a chronological overview of the advancements in the field, review of the UC Berkeley paper, explanation of relevance of tholin formation and hypothesized adaptations required to make NMR Spectroscopy on Titan possible.**

***Keywords*: μ metal, Analytical techniques, Nitrogen buffer gas, NMR Spectroscopy, No-field NMR, PHIP, Pump and probe lasers, Tholin, Titan, Zero-Field NMR.**

1. Introduction [2]-[15]

A. Basic Principles of No Field NMR

Nuclei contain both positively charged protons and neutrons which have no charge. Due to the concentration of the protons, nuclei are generally electrically charged. Most nuclei also have a characteristic spin which is denoted by I. The value can be integral, fractional or zero[] The spin of the electrically charged nuclei causes all the positive charges (protons) to align in one direction, and therefore form a magnetic field. The spin-magnet creates a magnetic moment proportional (μ) to the spin (I).

The spin causes the nuclei to gain angular momentum due to their circular, fixed motion. The angular momentum (determined by I) and the gyromagnetic ratio(ϒ) of the particle determine the total magnetic moment. The formula is: $\mu = \Upsilon * I$.

The protons in the nuclei exhibit spin-flip at a particular frequency called resonant frequency, under the influence of an external magnetic field (the strength of the field is represented by B_0), which can also be calculated using these values. For NMR spectroscopy, the induction of spin-flip is crucial; this requires the protons to jump to a higher energy level and the absorbance is only possible at this frequency. The formula for the resonance frequency is: $(\Upsilon * B_0) / 2\pi$.

NMR is used in pharmaceutical science to study pharmaceuticals and drug metabolism.

A magnetometer is defined as an instrument which is used to measure the direction and the strength of the magnetic field. The magnetism depends on various factors, for example the earth's magnetic field.

An atomic magnetometer comprises a glass cell containing vapor of alkali metal atoms. Using a resonant laser beam, they are optically pumped to give polarization on the order of unity. To measure the magnetic resonance frequency of the atoms in polarized state, they're probed using a laser which is directly proportional to the local magnetic field strength.

Unlike the inductive detectors whose sensitivity degrades at lower frequencies, an atomic magnetometer maintains its sensitivity and very low frequencies. Therefore, atomic magnetometers are employed as detectors of magnetic resonance at ultra-low-level fields, from 0 to 50 microTesla. This allows construction of NMR spectrometers without superconducting magnets, making them cheaper and more portable than conventional devices.

Alkali vapor-cell magnetometers are one of the most sensitive magnetic sensors, and contrary to inductive coils, their sensitivity is mostly independent of the frequency from the signal which renders them highly viable for low-field NMR and MRI applications.

Vapor-cell magnetometers are relatively economical, low-power, low-maintenance and resistant to temperature, vibrations and pressure and can be microfabricated as monolithic systems. This shows their usefulness as high-sensitivity, portable magnetic sensors and in compact sensor arrays.

Alkali vapor-cell magnetometers observe the resonance frequency of atomic spins to measure the magnetic field strength. Alkali spins can be hyperpolarized by optical pumping which enhances the sensitivity, making it easier to measure the precession frequency since it is optically measured by the

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Faraday rotation of an orthogonal beam or beam absorption. The precession of a group of polarized spins is used to measure the magnetic fields; the polarized state is brought about by exciting an optical transition which is connected to the atomic spin state.

B. History of NMR

Imaging and spectroscopy of NMR calls for new magnet configurations which need the conductor of the magnet to be placed inefficiently. High temperature superconductors have a high current-carrying capacity at very high fields, and with low cooling requirements in comparison to metallic superconductors however they do suffer from difficult processing and quench-protection techniques. Their use in high magnetic fields in large structures might lead to strength problems.

NMR is an essential technique for distinguishing proteins and other complex materials; the stronger the magnetic field, the more detailed the spectroscopy can be. The most powerful NMR magnets to date have a limit of 24 Tesla because of the materials they're made of. High temperature superconductors are used to get to higher fields for NMR magnets since they don't face the same restraints as the conventional NMR magnets.

High temperature NMR relying on inductive heating was introduced by Kendrick and Yannoni in 1990. Their setup had a paint coated glass tube with platinum suspension which was annealed (heat (metal or glass) and allowed it to cool slowly, in order to remove internal stresses and toughen it) at 830K for about 90 seconds. A heating coil was used to produce the necessary rf to heat the metallic layer; the eddy currents induced by the rf carry out the heating. The sample is heated by the transfer of heat from the metallic layer to the tube allowing the temperature to reach up to 730K in static NMR experiments. Since the source of the heat is localized, cooling of the sensitive equipment is easily accomplished.

For decades, superconducting magnetometers relied on low Tc helium-cooled superconductors until high transition temperature superconductors which operate on liquid nitrogen were introduced. Though the applications of the Tc helium-cooled superconductors are diverse, their use had been constricted due to the costly nature of helium cooling.

The main issue regarding high Tc superconducting magnetometers is that they produce a huge amount of noise.

C. Spectroscopy Principles

The NMR (Nuclear Magnetic Resonance) spectroscopy is widely used to identify different organic compounds. It can easily distinguish between different organic compounds and identify the different functional groups present. Organic molecules mainly consist of protons and carbon atoms by observing the chemical shift. Due to the chemical shift the nearby atoms in the organic compound will also experience the shift. Therefore, small organic molecules can be easily identified by matching the proton and carbon shifts in the molecule and comparing it to the predicted shifts.

If there is a match between the observed shift and the predicted shift the compound is identified to be the best possible match. If the shifts don't match the best matches tend to be the ones that are similar or close to the predicted value.

D. Spectroscopy Examples

There are many ways by which we can identify the organic compound. Some look up the observed shifts online in order to identify their compounds; some tend to use a commercial software which has its own NMR shifts (like the Cheomax NMR suite) to identify their compounds. There has been a spectral database that has been maintained by The National Institute of Advanced Industrial Science and Technology (AIST) in Japan for organic compounds (SDBS, http://sdbs.db.aist.go.jp/sdbs/cgi-bin/cre_index.cgi). This particular database contains six different types of spectra.

There are many uses of NMR (Nuclear Magnetic Resonance) spectroscopy, one of them being in pharmaceutical manufacturing. In order to develop a pharmaceutical product, it needs to go through three different phases. In the discovery phase (first phase) they attempt to find any potential leads, chemical compounds are screened against the receptors. In the second phase or the development phase the leads found in the first phase are tested again and attempts are made to formulate the product to prepare for the last step (last phase) which is the manufacturing of the product. NMR (Nuclear Magnetic Resonance) is used throughout these phases.

NMR analysis is present in the solution during the discovery for the illumination of the candidate molecules. Quantitative NMR is also used in this particular stage. In the development phase (second phase) NMR analysis is used for illumination from impure molecules, degraded products and metabolites present in the lead molecules. In the manufacturing process NMR is used mainly for identification, configuration and the purity of the substance used in the making of the pharmaceutical product including the pharmaceutical product itself.

E. Atomic Magnetometer

An atomic magnetometer comprises a glass cell containing vapor of alkali metal atoms. Using a resonant laser beam, they are optically pumped to give polarization on the order of unity. To measure the magnetic resonance frequency of the atoms in polarized state, they're probed using a laser which is directly proportional to the local magnetic field strength.

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2. Review [16]-[35]

A. Beating Squids

Superconducting Quantum Interference Devices (SQUIDS) utilised super conductors and functioned as magnetometers. While SQUIDS were the most sensitive magnetometers, they posed several disadvantages, primarily their requirement of cryogenic temperature. This posed a serious obstacle to the development of NMR technology, especially in regards to space exploration, since portability was compromised.

Atomic Magnetometers or Alkali Vapour Magnetometers have not only demonstrated greater sensitivity than SQUIDS by detecting below 1fT/√Hz, but they're less restricting in other aspects as well. Latest atomic magnetometers have dimensions in mm and show potential for further reduction in size. This is very appealing in the context of space exploration since this means the magnetometers would consume less space and be more light weight- ideal for attachment to probes.

More importantly, atomic magnetometers do not employ superconductors. Instead a combination of techniques including the use of mu metals, principles of pump and probe spectroscopy and shimmying coils are used. Additionally, the experiment adds PHIP which intensifies the "signal" and reduces the sensitivity required. Thus, the need for sub-zero temperatures is eliminated and NMR spectroscopy increases in its scope.

B. Cryogenic Temperature

Cryogenic temperatures are not used since atomic magnetometers don't use superconductors but rather use a pump laser to polarize the atoms. This makes the low temperature requirements for the superconductors to function redundant.

The SERF magnetometer is an alkali metal atomic magnetometer. It is an optical magnetometer which operates in a mechanism where the orientation of atomic spins do not get scrambled. It is considered as an optical magnetometer due to its use of the pump-probe full optical structure. The SERF magnetometers have a high sensitivity and don't need cryogenic temperatures to function, making them viable in fields where SQUID cannot function.

C. μ-metal

μ-metal is a soft ferromagnetic alloy. It has a high magnetic permeability thus it is used to shield the equipment from the Earth's static or low frequency magnetic fields. Furthermore, a layer of a ferrite shield is used to block high frequency magnetic fields and for the frequencies which are reduced to the μG level, they can be reduced by a set of 3 orthogonally placed shimming coils. The shimming coils reduce the magnetic field inhomogeneities (refers to the uniformity of the magnetic field) and optimise the image quality for the spectroscopy results. (static what) (why must it block) (how do ferrite shields work) (what does muG mean) (how do shimming coils work).

Using ferrite shields and four layers of μ-metal to counteract the influence of the Earth's magnetic field and other low-frequency fields, the magnetometer is shielded by peripheral frequencies which would affect the accuracy of the readings.

μ-metal is a ferromagnetic nickel-iron alloy which exhibits a high magnetic permeability and absorption loss at low frequencies. Since the μ-metal gets easily saturated due to its high magnetic susceptibility and low magnetostriction, it is most effective at blocking low frequencies. This blocking effect essentially diverts the magnetic field away from the rubidium vapour cell as the μ-metal's absorption ensures that low frequencies do not reach the cell.

D. Nitrogen Buffer Gas

Nitrogen acts as a buffer gas in the rubidium vapor cell. Along with ensuring a suitable environment inside the cell by maintaining pressure and preventing reaction (as it is an inert gas), the nitrogen buffer gas also impacts the sensitivity of the magnetometer itself. As the magnetometers progressively decreased in size to increase their scope and functionality, it was accompanied by reduction in sensitivity. However, by using buffer gases and maintaining certain levels of pressure, this problem could be combatted.

The use of buffer gases demonstrates multiple changes in the readings of the nonlinear magneto-optical resonances by increasing the frequency tuning range of the magnetometer. The peaks are significantly narrow, which changes the shape of the spectra, and the visibility is also altered due to the phenomenon of Electromagnetically Induced Transparency (EIT). These differences are explained by the ability of the buffer gas to change the interaction of the rubidium atoms. The buffer gases help to preserve the coherence of the resonance and reduce the wall collisions of the atoms.

E. Optical Pumping

The rubidium magnetometer uses optical pumping to excite electrons into a higher energy state, which is a method of artificially amplifying the nuclear spin polarization.

Two perpendicular pump and probe lasers record the vertical component of the sample's magnetic field, while the two beams (higher intensity pump beam and lower intensity probe beam) and the field intersect they are all orthogonal to each other.

The non-equilibrium, stretched state of the rubidium atoms caused by the pump laser and the effect of the magnetic field which further rotates the atoms together polarizes the atoms and aligns them at a specific angle. After a short time frame, a pulse from the weaker probe laser is used to identify the changes. The detected polarization is reflective of the magnetic field of the sample.

F. PHIP

Due to the lack of an external magnetic field, the protons would not naturally jump to a higher energy state, which would lead to unclear and irrelevant results. NMR spectroscopy is at the forefront of analytical techniques, however, these results could have been provided via other cheaper and simpler methods, thereby eliminating the functionality of NMR spectroscopy.

To be able to receive consequential results from no-field NMR spectroscopy, we need the molecules to be hyperpolarized. Once hyperpolarized, the NMR signals (which we aim to measure) become more intense. Since the signals are more pronounced, the magnetometers can receive them easily with a lower sensitivity. Fluids and gases are hyperpolarized using the technique of Dynamic Nuclear Polarization (DNP). PHIP is a para-hydrogen isomer with signal enhancement capabilities, as it can induce hyperpolarization due to its singlet state.

3. Applications [36]-[42]

A. Titans & Tholins

Titan is the second biggest moon in the solar system, and significantly different due to its dense and nitrogenous atmosphere. Due to its atmosphere, which contains the organic gas methane and the presence of tholins- complex polymers which are formed by the irradiation of abundant ices or gases.

Tholins are considered "chemical precursors to life", not only would they provide valuable insight regarding Titan, but they may help scientists understand the origin of life itself. The Cassini spacecraft utilized various spectrometers and revealed important information based on data collected via fly-bys.

However, we propose that for a more detailed understanding of the planet and the formation of tholins, we must use NMR Spectroscopy as well. With the recent advancements in the field, no field NMR Spectroscopy has become a valid option to use in the field of astrobiology. For a thorough investigation, it would be best to leave the spectrometer on the planet for a significant duration so as to monitor tholin formation.

Titan, due to its distance from the Sun and hazy atmosphere, has comparatively lower light intensity- roughly a hundredth of the light intensity on Earth. Therefore, solar energy is less effective. However, no field NMR Spectroscopy requires a small amount of power which could be easily provided by solar panels covering an area of $1m^2$.

The magnetometer reviewed in the earlier sections of this paper requires minimal power to function. While the total energy consumption is subject to change, during tuning for example, a working magnetometer only requires approximately 15mW for the pump laser and 5mW for the probe laser. As specified before, there is no magnetic field required and hence, no electricity is needed for inducting a magnetic field using an electromagnet. Similarly, temperature control is also unnecessary. The atomic magnetometer also utilizes buffer gas, mu metal, ferrite shields and shimming coils. Of all these components, the only one which has a power requirement is the shimming coil which is used for active shimming. Since, there is no significant variation of other frequencies on Titan, the coils can be replaced with ferromagnetic pellets which provide passive shimming.

On Titan, covering $8\times(10^{12})m^2$ with solar panels would generate about $1.36\times(10^6)$MW of power. The pump and probe lasers use a total of 20mW of power, thus to produce this amount of power, we would need a solar panel with the area of $((20mW/1.36\times(10^6)MW)\times(8\times(10^{12})))m^2$. So, a solar panel of approximately $1.2cm^2$ is required. We use a $1m^2$ solar panel to account for unprecedented drops in light intensity so the device runs consistently.

The efficacy of solar panels does vary as Saturn, and consequently Titan as its synchronous moon, revolve around the Sun. One revolution takes place in 29 years. Using data from the Cassini spacecraft (2007-2014), we observed an 18.6% decrease in solar power over the course of 13 years. Yet, this would have negligible effect on the magnetometer because of its low power consumption and small time frame of maximum 5 years.

Another variable which must be accounted for is accumulation of tholides on the surface of the solar panels. This can be easily countered by using self-cleaning solar panels, which use nanotechnology to ensure cleanliness. There can be upto a 40% drop in power capabilities due to a unclear surface but the panels prevent it. Only a small area ($1m^2$) of panels is required, so there is a high feasibility.

4. Conclusion

This paper presented an overview on extension of advances in zero-field nuclear magnetic resonance spectroscopy to monitor tholin formation on titan.

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