Nicole-Ann Castillo

PHYS 134

Group 2

Lab 1: Nuclear Magnetic Resonance

Introduction

Nuclear Magnetic Resonance (NMR) occurs when a nucleus of some atom is encouraged to spin due to a surrounding magnetic field, first observed by Isidor Rabi in 1938 (Becker, E., 1993). The nucleus behaves like a spinning dipole magnet, thus possessing angular momentum and a magnetic moment. This phenomenon is best described with quantum mechanics. A proton has a spin of ½, meaning that it has two possible energy states. When immersed in a magnetic field, the particle will precess at a certain frequency in order to align parallel (or anti-parallel) with the field. This precession frequency v is called the Larmor frequency, given by

$$hv = \Delta E \tag{1}$$

where ΔE is the energy separating the two energy states, and h is Planck's constant. For a sample in thermal equilibrium, there is an excess of protons in the lower energy state parallel to the magnetic field, meaning there is a slight net magnetization induced on the sample (Feynman, R. et al., 1963). We can equalize the populations occupying these energy states, however, by sending a radio frequency (RF) pulse at the Larmor frequency. This causes a non-equilibrium population of energy states that will decay back to equilibrium once the RF signal is removed, resulting in a free induction decay (FID). The time constant for this decay is known as the spin-lattice relaxation time T_1. It can also be referred to as the longitudinal relaxation time, since

it determines how long it takes for the spins to relax in the direction of the external magnetic field. We can express the decay with an exponential equation

$$A(t) = A_o \left| 2e^{(-t/T_1)} - 1 \right| \tag{2}$$

where A_o is the amplitude of the highest peak of the pulse, at 0 delay time t. There is also a transverse relaxation time, also known as the spin-spin relaxation time, T_2, which describes the time of decay along the axis perpendicular to the external magnetic field. This will also take on the form of an exponential decay equation

$$A(t) = A_o e^{(-t/T_2)} \tag{3}$$

We can essentially rotate the axes at which we observe the spins of the protons in a sample. Usually, transverse relaxation time, and NMR in general, is used in medical applications such as brain mapping (Deoni, S., 2009) and other MRI's (Hazelwood, C. et al, 1974). The goal of this lab is to find both T_1 and T_2 for a sample of mineral oil and see how they compare to each other. We will first find the magnetic field gradient by observing the FID signal after a 90° RF pulse, and then use the spin echoes to obtain values for the relaxation times.

Methods & Materials

We will be performing a series of experiments to estimate the relative magnetic field gradient, T_1, and T_2. Our set-up consists of the Pulsed NMR Spectrometer, the permanent magnet that the sample is embedded in, and the oscilloscope to show our readings. By now we are familiar with the oscilloscope. The permanent magnet mainly needs to be connected with the Pulsed NMR Spectrometer, with the vial of mineral oil inside of the slot between the magnet disks. Figure 1 below shows a simplified version of how all of the equipment is connected

together. In the following subsection, we will detail the set up for TeachSpin's Pulsed NMR Spectrometer, since it required the most attention.
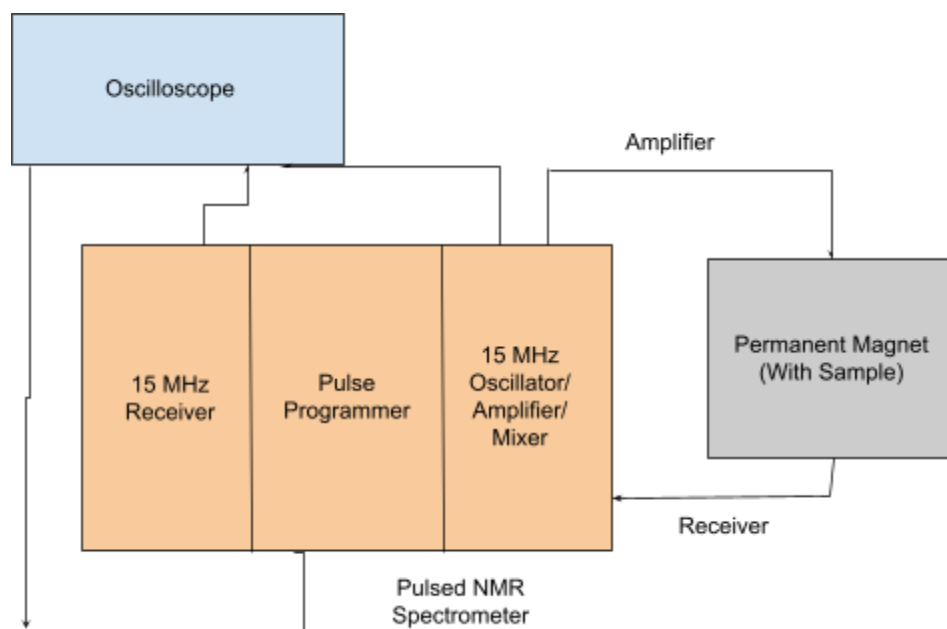
Figure 1: A simple diagram of the general set up for the experiments. Each of the connections will be made with provided wires, and the arrows show the directions the signals are being input or output from each device. The Pulsed NMR Spectrometer will have three operation bays with their own function, so it is important to know which section has a certain control.

As we can see from the diagram, the permanent magnet will receive a pulse from the spectrometer, which will also receive a signal from the magnet with the sample. This signal, and an averaged signal, is then displayed on the oscilloscope, which we will expand on later.

The Pulsed NMR Spectrometer

This device is what requires the most set up. There are three main bays of the Pulsed NMR Spectrometer: the Receiver, the Pulse Programmer, and the Amplifier/Mixer. The Pulse Programmer is where we set up the pulse we send to the magnet. We can change the number of pulses, their axes, and the delay times on this bay. The Amplifier/Mixer connects us to the

permanent magnet, and is where we can change the frequency of our pulse. The Receiver obtains the signal from the pulse after it is sent to the permanent magnet, which will display on the oscilloscope in two channels. In Channel 1, DETECTOR OUT shows the average signal amplified output from the signal to the permanent magnet, whereas Channel 2 will show MIXER OUT, which will help us precisely tune the Larmor frequency. Figure 2 shows these connections on the spectrometer, since it is very important to connect the right bays for the desired signal.
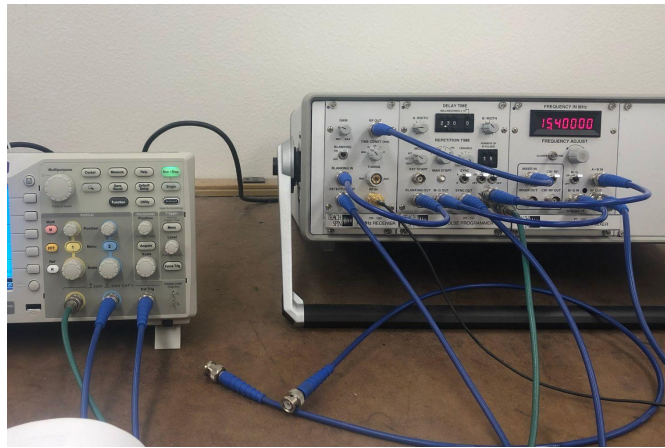
a)
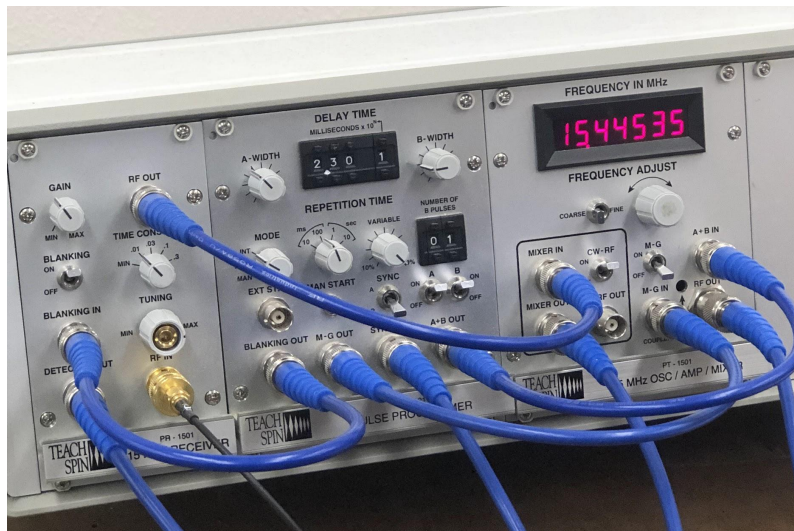
b)

Figure 2: The set up for all equipment. The oscilloscope will mainly be connected to the spectrometer as shown in **a)**, and spectrometer will also be connected to the permanent magnet through the receiver and amplifier. Image **b)** has a closer view of these connections on the NMR spectrometer. Note that the frequency and delay times shown in the image are not the ones we took data of, as they change frequently throughout each experiment. However, the pulse programmer always has MODE set to "Int.", VARIABLE at 100%, and REPETITION TIME at 100 ms. On the receiver, TUNING should be at the maximum and GAIN should be around 30%, with the time constant at 0.01.

1. **Free Induction Decay (FID)**

   With this experiment, we want to estimate the relative magnetic field gradient by observing the FID of our sample. We apply a 90° pulse by adjusting the A-width knob on the pulse programmer bay so that the oscillator shows the maximum amplitude of the peak in Channel 1. This should be at about 20% A-width. As stated earlier, the pulse will produce an oscillating signal at the precession frequency that will decay over time, until the pulse ends. Figure 3 below shows what the signal should look like, once adjusted to take data.
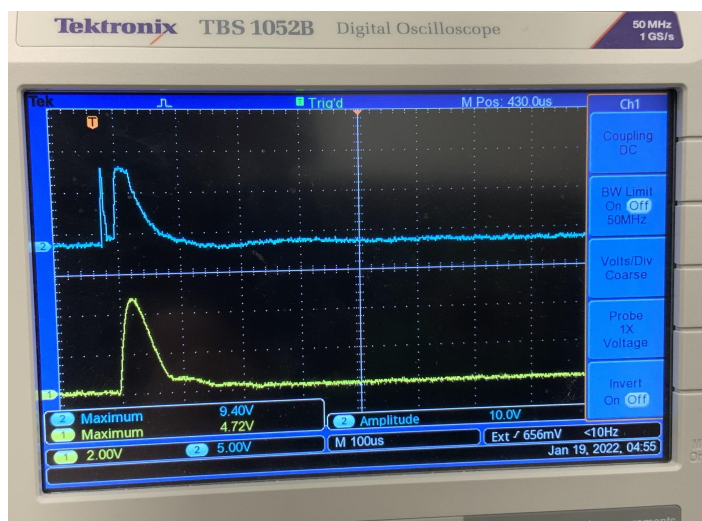

Figure 3: Oscilloscope showing Channel 1 (yellow) and Channel 2 (blue) readings from each input. Initially Channel 2 will have a number of beats within a pulse, but for more accurate readings of the amplitude we do not want any beats from the signal. This is adjusted by changing the frequency on the amplifier bay of the

spectrometer. The ideal precession frequency will have no beats on this signal. We can also adjust the A-width knob to see how it changes the amplitude of the signal in Channel 1. A full maximum signal will be a 90 ° pulse and consequently, a 180 ° pulse will give a 0 signal.

2. Spin-Lattice Relaxation Time (T_1)

To measure T_1, we set the A-width to emit a 180 ° pulse, which should be a 0 signal. We set a delay time t on the pulse programmer, and apply a 90 ° pulse with the B-width knob. From the resulting signal on the oscilloscope, we measure the amplitude on Channel 1. The B-pulse ticker should have a "1" displayed, meaning we get one B-pulse to take readings from.

3. Spin-Spin Relaxation Time (T_2)

For this sub experiment, we will use three different methods of varying accuracies to find a reasonable value for T_2. Note that once we set the A and B pulses to the correct spins, we do not have to change them for collecting the rest of data for each method. We should only have to adjust the delay time t on the amplifier/receiver. This is because the Pulsed NMR Spectrometer already knows what to do for these processes and the oscilloscope will show the result of such adjustments. We record the amplitudes of this peak for different delay times t. From the data, we should start to see a decaying pattern as t increases.

a. The Hahn Method

The Hahn Method, or the two-pulse spin echo method, employs exactly what it says in the name. This time, our A-pulse is set to 90 °s about the y-axis, and the B-pulse is set to 180 °s about the y-axis. Compare this to the method to find T_1, which had a 0 signal for A at 180 °s and

a 90 ° B pulse. And similarly to finding T_1, we take data for varying delay times $t = \tau$ where the

echoes will appear. Notice that, as delay time increases, the amplitudes will decrease to 0 and

start to increase again. This reflects in equation **[2]**, where the absolute value keeps the amplitude

from reaching a negative value.

b. The Carr-Purcell Method

The Carr-Purcell Method uses the same steps as Hahn, but instead we will use a multiple

pulse spin echo sequence. We increase the number of B pulses to "2" on the spectrometer so the

readout on the oscilloscope should show spin echoes at τ and 2τ. This means we should take data

for two peaks that show up on the oscilloscope.

c. The Meiboom-Gill Method

The Meiboom-Gill method also employs the multiple spin echoes, but we will shift rotations

from about the y-axis to the x-axis. This reduces possible internal error with the 180 ° pulse that

the Carr-Purcell method does not take into account. Hopefully, this will give us a clearer value

for T_2. We shall record data in the same manner as we did for the Carr-Purcell method. Figure 4

shows the difference in axes between the Carr-Purcell and Meiboom-Gill methods, since they
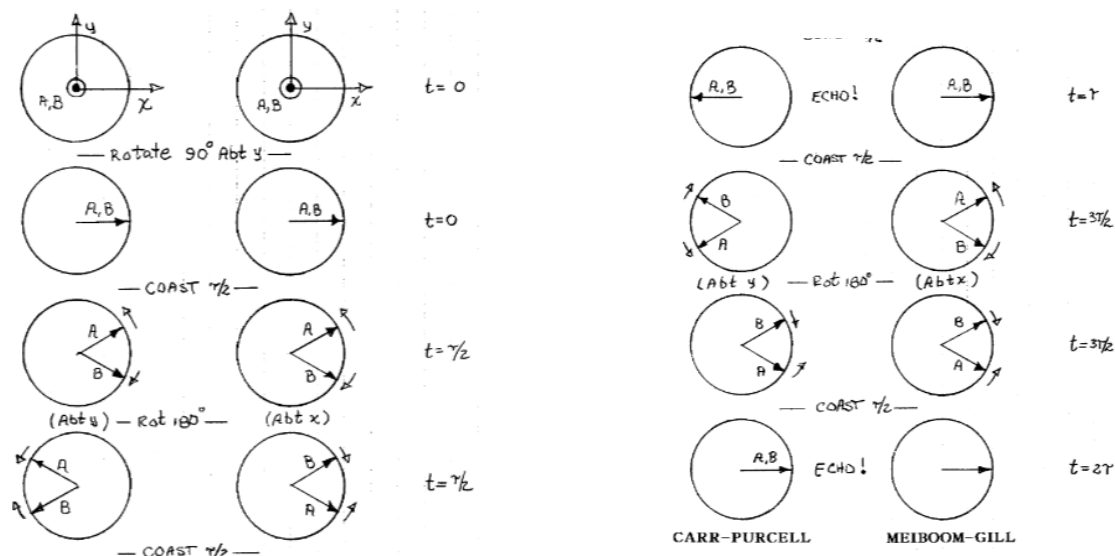
only differ in orientation.

Figure 4: A visual representation of the spins with A and B pulses applied. Carr-Purcell is in the left column and Meiboom-Gill is on the right. Note that there isn't much of a difference in the data taking process for these methods. Once you set the pulses to the correct width with the knobs, the spectrometer will know what to do. (*PHYS 134 Advanced Laboratory*, 2022)

Results & Error Analysis

Overall, we find that the amplitudes from each experiment show an exponential decay over time, which is agreeable with the patterns of NMR as nuclei realign themselves with the surrounding magnetic field. The FID experiment shows that the magnetic field gradient $(\frac{1}{B})(\frac{dB}{dx})$) results is an exponential decay, shown in Figure 5.
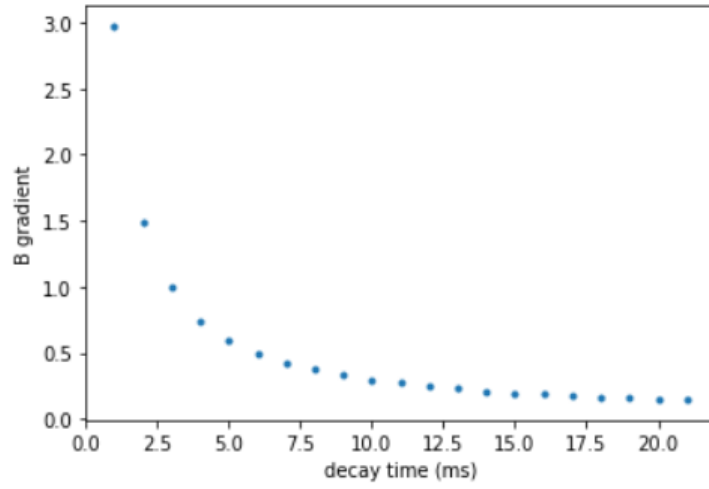
Figure 5: The magnetic field gradient plotted with respect to time. We are given the sample size and time constant corresponding to time t.

For T_1, we fit the generated plot of amplitude with respect to time in Figure 6, and fit a curve to find a value that corresponds to equation **[2]**. We find T_1 to be around 30 ms for the sample of mineral oil.
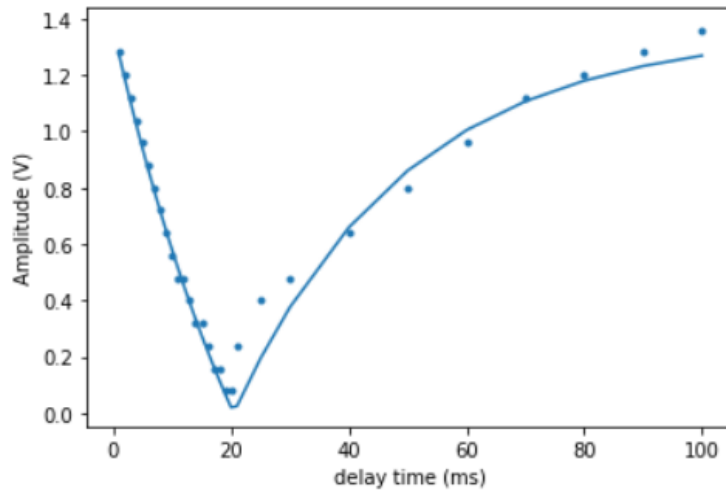

Figure 6: Amplitude of the peaks from Channel 2 over a delay time t. As discussed earlier, we assumed the absolute value from equation 2 and the sinusoidal nature of the magnetic field with a pulse would generate a plot that has a decreasing value to 0 that would increase again. The right side is a less steep incline than the left.

We are most interested in T_2 since it reduces interference from the inhomogeneities from the magnetic field. The three methods we used generate the plots shown in Figure 7. As predicted, the methods are quite close in results, or at least, in the decay shape from the data because of the slight adjustments in technique. Carr-Purcell added more pulses from Hahn, and Meiboom-Gill changed the axis of the spins. The plots show an exponential decay, and assuming we follow equation (#), we find the values T_2 are 1.062 from Hahn, 21.79 (echo 1) and 26.25 (echo 2) from Carr-Purcell, and 21.413 (echo 1) and 24.45 (echo 2) from Meiboom-Gill.

a)

b)
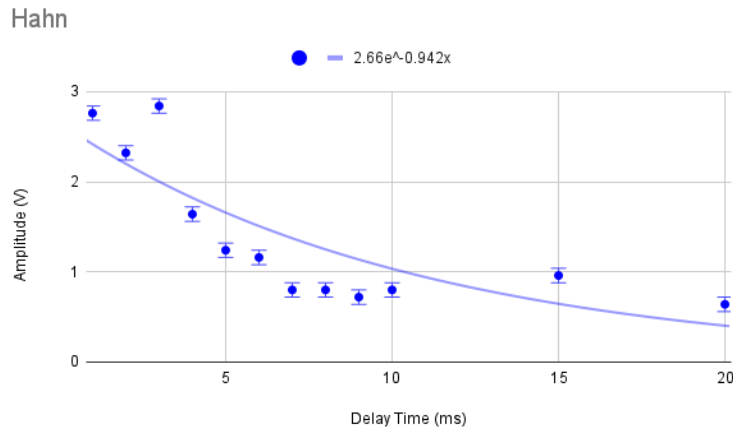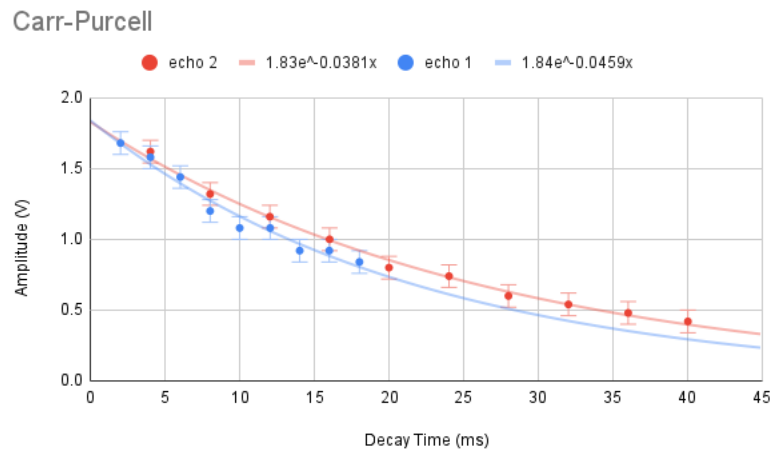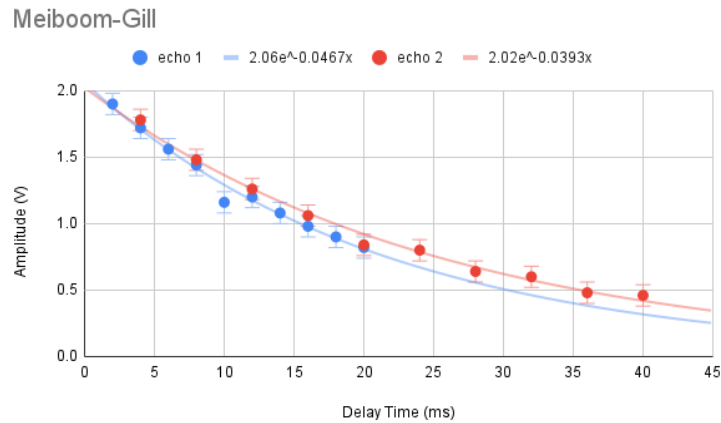
Meiboom-Gill

c)

Figure 7: Plots of amplitude with respect to delay times for a) Hahn b) Carr-Purcell and c) Meiboom Gill. Though they each have a decaying trendline. Hahn is far more scattered than the last two methods. This could be because the Hahn method considers the particles to be more or less stationary, causing a reading to have a faster diffusion rate. Carr-Purcell and Meiboom-Gill both take this into account with the multiple spin echo method. Both of these methods have closer readings to their trendline, given the margin of error. We see that in those two plots, the 2nd echo is at a time double from the first one, so the data points effectively shift to the right and their results for T_2 are a bit larger.

For the last experiments, we have concluded that the standard deviation is 0.080 V. Since we have recorded Channel 2 with an oscillating signal at a minimum and maximum value, but we recorded from Channel 1, which was the average reading of the signal. We have derived this deviation from other readings, so in another trial of this experiment it would be better to record Channel 2 readings for a more accurate telling of the margins of error. For now, though, this will do since they had the same oscillating pattern. We can also note that, over time, the permanent magnet is also affected by the changing temperature from the environment, so the precession frequency would change ever so slightly. This means that we had to change the spectrometer a few times over the course of the experiment. But since we are finding time constants, this should not be a huge factor in our final results as long as we have a signal with as few beats as possible.

Discussion

In this lab, we observe nuclear magnetic resonance with a sample of mineral oil embedded within a permanent magnet. We find the magnetic field gradient via FID, with a time constant of 33 ms. This is synonymous to our value for the spin-lattice relaxation time of 30 ms, and our found values of spin-spin relaxation time from the Carr-Purcell and Meiboom-Gill methods. Each of our plots show an exponential decay, which is due to the RF pulse disappearing from the external magnetic field as the spins realign to equilibrium.

Though we have reasonable results that are shown qualitatively with the graphs, there is still uncertainty with the exact relaxation times obtained from the experiments. Since this was our first experience with this device, we were also uncertain on how to accurately obtain the data and had the most trouble with making sure we had the correct setup. Taking the data required some trial and error, and we hope that in future experiments there will be an opportunity to better understand how the equipment can read out the most accurate results.

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