Rooftop End Mirror Automation: A Quasioptical Improvement to Increase the SNR in EPR Spectroscopy

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Abstract

Scientists in various disciplines, from physics to food science, use electron paramagnetic resonance (EPR) spectroscopy to identify parameters of paramagnetic materials. To perform EPR spectroscopy, linearly polarized microwaves irradiate a paramagnetic sample in a modulated magnetic field. The sample partially absorbs the microwaves when the resonant condition is satisfied, which changes the microwave polarization. After absorption, a series of parabolic mirrors reflect the resulting microwaves back toward their source. In induction mode, the microwaves perpendicular to the initial microwave polarization are reflected by a polarizer and are then detected as an EPR signal. However, imperfections throughout the quasioptics also change the polarization, necessitating a way to correct unwanted changes in polarization. The solution is to include a tunable rooftop end mirror within the sample holder. However, manual rotation of the rooftop mirror results in imprecise optimization and inefficient data acquisition. Implementing an automated rooftop end mirror system, including a step-servo controlled by LabView with serial communication and a 3D printed connector with timing pulleys, increases the angular precision which improves the SNR in EPR spectroscopy by decreasing the baseline EPR signal before amplification to 1% of the baseline value.

Introduction

A versatile, noninvasive tool for determining the properties of paramagnetic samples by analyzing unpaired electrons' spin is found in electron paramagnetic resonance (EPR) spectroscopy. Because of the multitudes of information its analysis can provide, EPR spectroscopy is used by a variety of scientists. For example, biologists can learn more about proteins tagged with paramagnetic material. [1] Chemists and physicists can study oxidation to improve batteries. [2] Food scientists can examine free radicals in food to improve taste and nutrition. [3] EPR spectroscopy meaningfully contributes to many fields and, therefore, is a tool worthy of improvement.

EPR spectroscopy delivers an information-rich absorption spectra. This spectra is obtained by taking advantage of the Zeeman effect. Unpaired electrons have a quantum property called spin which can be one of two eigenstates: +1/2 and -1/2. When there is no external magnetic field, there is no energy difference between the electrons in the spin-up and the spin-down states. Therefore, the electrons are in degenerate states. However, once there is an external magnetic field, the electrons in the spin-up state linearly increase in energy, and the electrons with spin-down linearly decrease in energy. The energy E is related to the g-factor g, the magnetic moment μ_B, and the external magnetic field B_0.

$$E = \pm\frac{1}{2} g \mu_B B_0 \tag{1}$$

Therefore, there is a difference in energy proportional to the magnetic field as seen in Figure 1. A 240GHz microwave passes through the sample to find this energy difference. This microwave will have a discrete amount of energy E where f is the microwave's frequency.

$$\Delta E = g \mu_B B_0 = hf \tag{2}$$

In EPR spectroscopy, spectra can be found in two ways: a constant magnetic field with a modulated microwave frequency or a constant microwave frequency with a modulated magnetic field. Our experiment modulates the magnetic field to obtain the absorption spectra. The absorption will be maximized when the energy difference between the spin states equals the energy of a photon in the microwave.

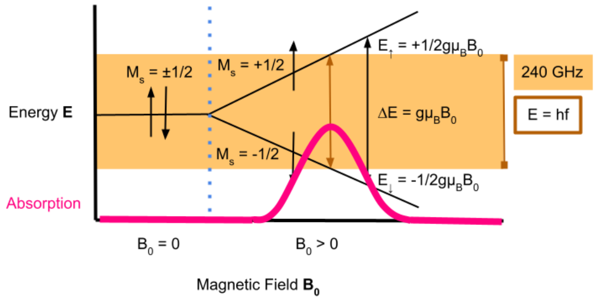


Figure 1: *EPR Spectroscopy with Resonance Condition. Microwaves (orange) with energy equal to the energy difference established by the Zeeman Effect are absorbed by the sample. The absorption spectra is seen in pink*

A wire grid polarizes the microwave before it makes its journey down the probe to the sample and back up the probe toward the wire grid as seen in Figure 2. Then, the wire grid reflects any perpendicularly polarized component of the microwave, which comprises the EPR signal, to the detector. Ideally, when there is no sample, the polarization of the microwave will not change so that no signal will be detected. However, imperfections in the quasioptics cause undesired changes in polarization. A rooftop end mirror is used in the sample holder to correct for these polarization changes.

Before, the rooftop end mirror was rotated manually in ITST's EPR Spectrometer. This tedious data acquisition method slowed down data collection and created an opportunity for improvement in the experimental process.

In this work we show that inplementing an automatic rooftop end mirror rotation system decreases the baseline signal to 1% of the original baseline signal and expedites the data acquisition process. With the improvement, a step-servo rotates to find the optimal position for the rooftop end mirror with feedback from the detector to minimize the signal when there is no sample. The step-servo is attached to the EPR probe using an adjustable 3-D printed connector that rotates the rooftop mirror with a system of gears and timing pulleys.

With feedback from the detector, the step-servo rotates the rooftop mirror to precisely minimize the baseline signal, therefore improving the SNR and efficiency of the experiment.

240 GHz ITST EPR Spectrometer with 40mW Low Power Source

A 240 GHz microwave with a 40mW low power source is used to irradiate the sample in ITST's EPR spectrometry experiment.

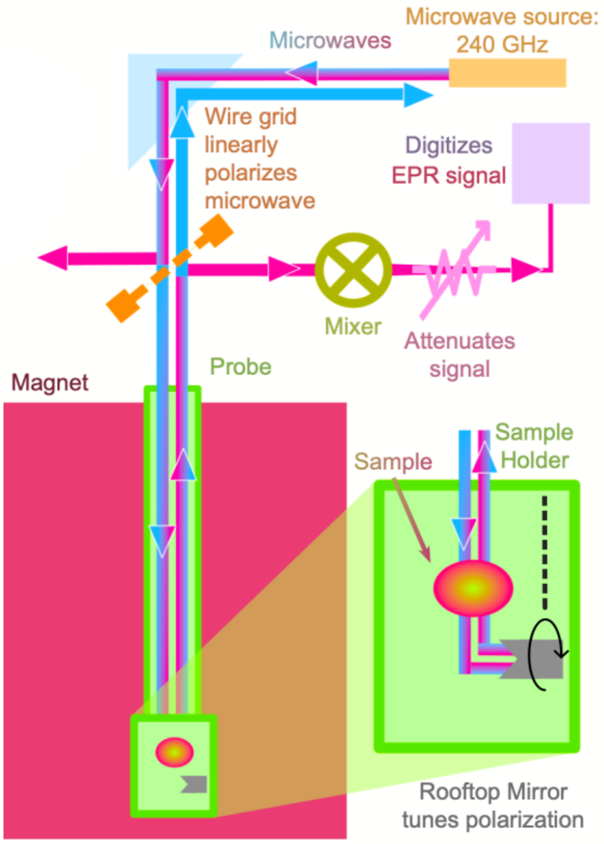


Figure 2: *240 GHz ITST EPR Spectrometer. Copolar microwaves (blue) are sent through and partially absorbed by the paramagnetic sample, producing crosspolar (pink) microwaves which comprise the EPR signal.*

As shown in Figure 2, copolar microwaves (blue) and crosspolar microwaves (pink) travel through a wire grid (orange). Copolar microwaves pass through the grid, but crosspolar microwaves are reflected. This allows the copolar microwaves to travel through the probe to the sample holder (green) which houses the sample and quasioptoics. Absorption by the sample and imperfections in the quasioptics change the polarization, resulting in crosspolar microwaves. The microwaves travel out of the probe back to the wire grid. The copolar microwaves travel through the wire grid as leakage, and the crosspolar microwaves are reflected toward a series a mixer, an attenuator, then finally an amplifier before the signal is digitized.

The polarization will change from copolar to crosspolar for two reasons: 1) absorption by the sample and 2) imperfections in the quasioptics. The polarization changes due to absorption by the sample is desirable, unlike changes due to imperfections in the quasioptics. These crosspolar microwaves compose the EPR signal. These imperfects therefore increase the magnitude of the measured baseline signal. Larger baseline signals require greater amounts of attenuation by the attenuator which adds additional noise to the signal. In order to optimize the signal to noise ratio in continuous wave EPR spectroscopy, it is essential to minimize the baseline signal by minimizing the polarization changes caused by the imperfection in the quasioptics.

Rooftop End Mirror

Unwanted polarization changes can be minimized by using a rooftop end mirror in the sample holder. The rooftop end mirror (roof mirror) changes the polarization of a microwave and reflects the wave back along the same path.

The geometry of the rooftop end mirror changes the polarization as a function of the mirror's rotation as shown in Figure 3. The mirror is an aluminum cylinder with two perpendicular faces cut into one end. These faces establish a mirror axis. Microwaves travel to the mirror and is reflected off the first face, then the second. Because the faces are perpendicular, the microwave will always be reflected back in the same direction. Immediately before the microwave is reflected off the first face, the linear polarization can be described as θ from the axis made from the intersection of the two mirror faces. The polarization can be separated into two components: parallel to the mirror axis and perpendicular to the mirror axis. As the microwave is reflected off the two faces, the parallel component remains unchanged, but the perpendicular component changes by 90° at each face. Therefore, the perpendicular component experiences a 180° reflection. After the roof mirror, the linear polarization will be $-\theta$ away from the mirror axis. Therefore, the roof mirror changes the linear polarization by 2θ. The change in polarization can be changed by rotating the roof mirror. Rotating the mirror will change the orientation of the mirror axis; therefore, the angle between the initial polarization and the mirror axis will change, which is directly related to the net change in polarization.

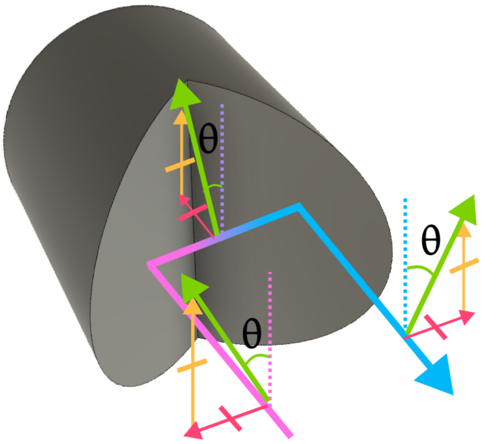


Figure 3: *Rooftop end mirror changes microwave polarization (pink to blue) by 2θ. The intersection of the rooftop mirror faces establishes a 'rooftop mirror axis' (the pink, purple, and blue dashed lines are parallel to this axis). Microwaves have an initial polarization (green) which is θ to the left of the 'rooftop mirror axis.' After the microwaves are reflected by the two mirror faces, the polarization with be θ to the right of the 'rooftop mirror axis,' therefore, the change in polarization is by 2θ.*

Improvements Possible with the Automatic System

Before the implementation of the automatic system, the knob was turned manually to minimize the signal at the detector. Additionally, the roof mirror is rotated by a series of 3D-printed gears connected to a knob at the top of the EPR probe. Because of significant gear backlash and the manual knob, the optimizing process was imprecise and inefficient. Automating the rooftop end mirror will ensure that the signal is minimized precisely and efficiently.

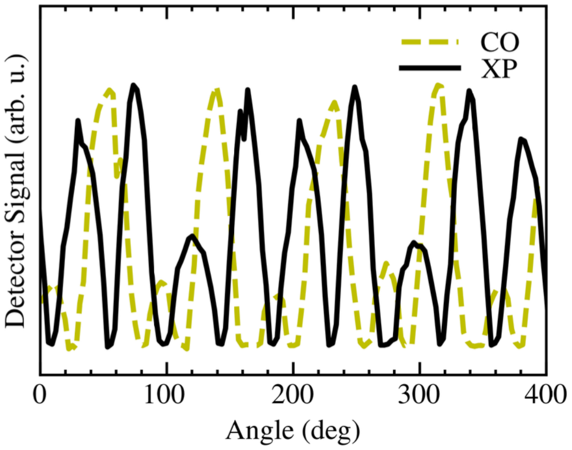


Figure 4: *Detector signal (arbitrary units) for copolar (CO) and crosspolar (XP) microwaves for different angles of the rooftop mirror with manual rotation. Manual rotation by hand causes imperfections while recording the signal at various angles. The copolar and crosspolar signals should be at their maximums while the other is at their minimums because of the rooftop end mirror geometry. If the signal polarization is manipulated to maximize the crosspolar signal, there will be little to no copolar signal to detect. [4]*

Physical Rotation System Connector

The rooftop end mirror automation system rotates an Applied Motion Step-Servo (TSM17Q-1RG) and receives signal feedback from the EPR detector to optimize the SNR ratio by decreasing the necessary attenuation value.

Connector Goals

The connector between the step-servo and the roof mirror top must be seamlessly integrated into the existing experimental setup. It must be compact and easily removable with the EPR probe to load samples. The most efficient design involves attaching the connector directly to the EPR probe, which is located close to the roof mirror knob. Additionally, it must be made with non-magnetic materials to avoid interacting with the magnetic field used for EPR spectroscopy.

Connector Construction

The connector is made of three main 3D-printed components as seen in Figure 5. The first component (dark gray) is a rectangular clamp with a square platform to house the step-servo. The robust design allows the servo to be firmly attached to the connector. The tall, vertical clamp design is used to limit the range of vertical motion. The second component (dark gray) is a rectangular arm inserted into the first piece and attached to another clamp. The third component (pink) is the other side of this clamp which firmly attaches the connector to the EPR probe. The rectangular arm and clamp allow the step-servo to be at an adjustable distance from the roof mirror knob while maximizing the clamping surface area. Both of the clamps are tightened by brass 4-40 screws and bolts. The components have hexagonal countersinks to house the bolts, which keeps the nuts stationary during small adjustments.

A 20-tooth timing pulley was added to the rotating rod of the step-servo to engage with an 11-inch timing pulley. The roof mirror knob is replaced with an additional 20-tooth timing pulley to engage with the same timing belt. The adjustable cylindrical clamp is used to shorten and extend the connector to engage the timing belt. The rooftop end mirror rotates with the step-servo because of the timing belt connection.

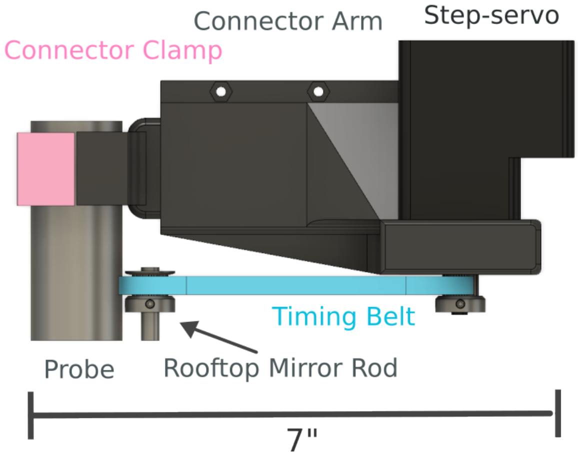


Figure 5: *Connector assembly design connects directly to the EPR probe so the step-servo can easily rotate the rooftop end mirror rod. The connector arm has a robust design to maintain its position while tension from the timing belt (blue) pulls on it.*

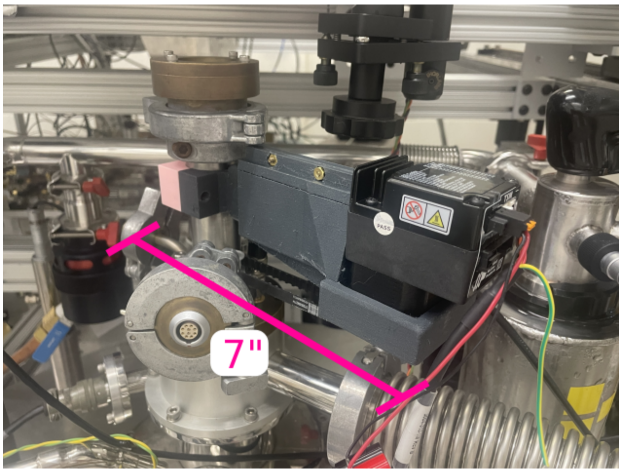


Figure 6: *Connector attached to EPR probe in EPR spectrometer. The length of the rotation system is 7 inches when extended to maintain tension in the timing belt.*

LabView Program

Driver

The rooftop end mirror automation system driver uses serial communication to rotate the step-servo. The driver is coded in LabView to easily integrate the system into the existing EPR spectroscopy experiment interface.

Responding to Voltage Feedback

Before integrating the system into the apparatus, we tested the system's ability to respond to voltage feedback by developing an intermediate apparatus. I created a circuit with a potentiometer which produced a voltage as a function of rotation, as seen in Figure 7. The circuit consists of a simple voltage divider and a potentiometer. The timing belt connects the potentiometer to the step-servo, so the voltage at V_{out} is a function of the potentiometer's rotation.

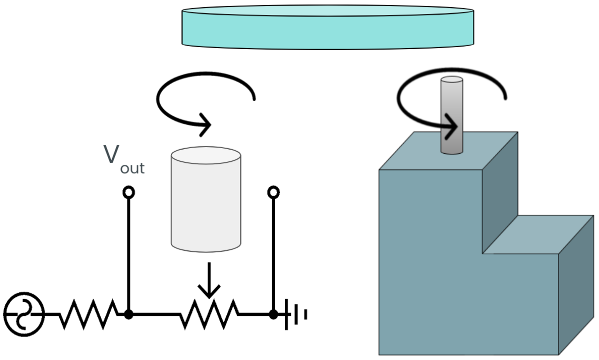


Figure 7: *Step-servo (gray-blue) rotates a potentiometer (light gray) using a timing belt (blue) to change voltage output of voltage divider (circuit diagram in black). With this configuration, voltage is a function of rotation, mimicking the rooftop end mirror in the EPR spectrometer.*

The *Find Goal Voltage* program compares the goal voltage to the output voltage. Depending on which one is greater, the program will tell the step-servo to turn which will change the resistance of the potentiometer. Therefore, V_{out} will increment towards the goal voltage. This is repeated until V_{out} is the same as the goal voltage.

This intermediate step provided a platform to troubleshoot the automatic rotation as a response to a voltage signal before moving into the EPR apparatus.

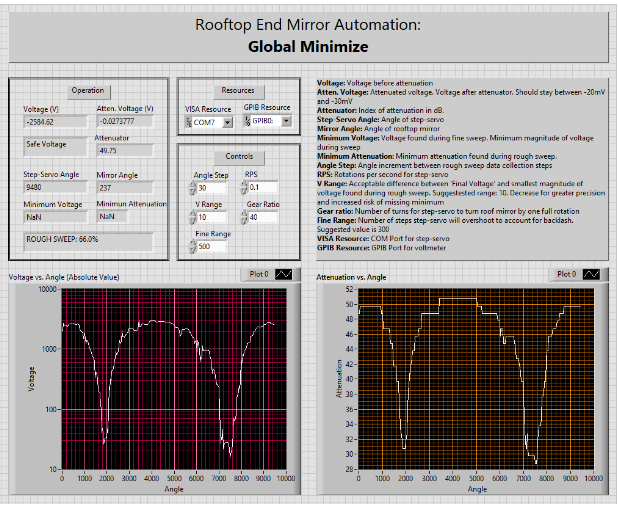
Global Minimize Program

The *GlobalMinimize.vi* finds and maintains the minimum voltage signal detected. It begins with an initial rough scan to obtain a signal magnitude for each degree of the rooftop mirror. The step-servo returns to the angle that corresponds to minimum signal and overshoots it to account for any gear backlash. Then, the step-servo will rotate the rooftop mirror by small increments until the voltage is within a specified range of the minimum voltage identified during the rough scan.

During this process, the voltage is attenuated to protect the amplifier which prepares the signal for digitization.

Program Outline

1. Adjust to proper attenuation (Atten. Voltage between -30mV and -20mV)
2. Rough Scan
   - Records and plots voltage (V) vs. step-servo angle (degrees)
   - Records and plots attenuation (dB) vs. step-servo angle (degrees)
   - Advances by *Angle Step*
   - Notes minimum voltage found
3. Returns to angle of identified minimum voltage and overshoots by *Fine range*
4. Fine Scan
   - Compares voltage to minimum voltage found during rough scan
   - If the voltage is within *V range* of minimum voltage found during rough scan, fine scan is completed
   - If not yet reached, advances by 5 step-servo degrees and repeats
5. Reports final *Minimum Voltage* and *Minimum Attenuation*



Rooftop End Mirror Automation:
Global Minimize

Operation
Voltage (V): -2584.62
Atten. Voltage (V): -0.0273777
Safe Voltage
Attenuator: 49.75
Step-Servo Angle: 9480
Mirror Angle: 237
Minimum Voltage: NaN
Minimun Attenuation: NaN
ROUGH SWEEP: 66.0%

Resources
VISA Resource: COM7
GPIB Resource: GPIB0:

Controls
Angle Step: 30
RPS: 0.1
V Range: 10
Gear Ratio: 40
Fine Range: 500

Voltage: Voltage before attenuation
Atten. Voltage: Attenuated voltage. Voltage after attenuator. Should stay between -20mV and -30mV
Attenuator: Index of attenuation in dB.
Step-Servo Angle: Angle of step-servo
Mirror Angle: Angle of rooftop mirror
Minimum Voltage: Voltage found during fine sweep. Minimum magnitude of voltage during sweep
Minimum Attenuation: Minimum attenuation found during rough sweep.
Angle Step: Angle increment between rough sweep data collection steps
RPS: Rotations per second for step-servo
V Range: Acceptable difference between 'Final Voltage' and smallest magnitude of voltage found during rough sweep. Suggestested range: 10. Decrease for greater precision and increased risk of missing minimum
Gear ratio: Number of turns for step-servo to turn roof mirror by one full rotation
Fine Range: Number of steps step-servo will overshoot to account for backlash. Suggested value is 300
VISA Resource: COM Port for step-servo
GPIB Resource: GPIB Port for voltmeter

Figure 8: *Graphical User Interface for 'GlobalMinimize.vi'. The interface includes many parameters so the user is given sufficient control over the automated process.*

Results and Discussion

With no sample inserted, the *GlobalMinimize.vi* was run and the following results were obtained for the EPR signal received for various angles of the rooftop end mirror.

After performing the global minimization program, the crosspolar signal reached a minimum of 0.12V compared to a maximum of about 70V, about 1% of the baseline signal as seen in Figures 9 and 10. This order-of-magnitude improvement is evidence of the success of the automatic rooftop mirror rotation system. With a significantly decreased baseline signal, the attenuator can operate at lower attenuation values, resulting in less noise in the signal.

Additionally, compared to manual rotation, automatic rotation improved the efficiency of finding the global minimum signal. Depending on the scan frequency, the *GlobalMinimize.vi* can run on the order

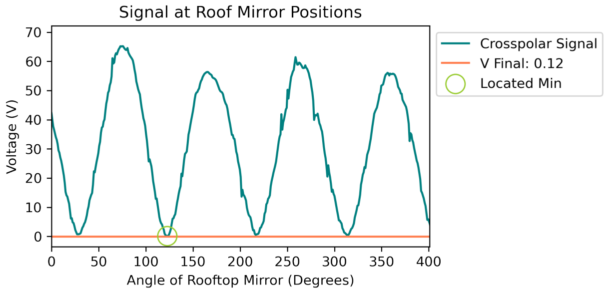


Figure 9: *Crosspolar signal for rooftop mirror angular positions with global minimum indicators. The green circle shows the voltage and minimum signal found during the rough scan. The coral line shows the minimum signal found after the fine scan.*

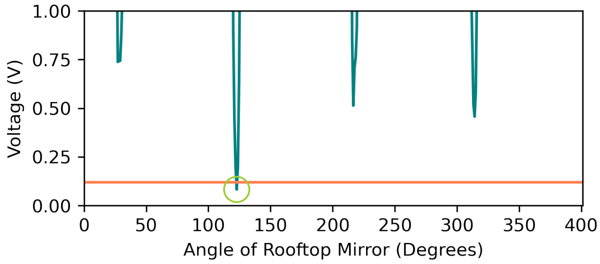


Figure 10: *Crosspolar signal for rooftop mirror angular positions with global minimum indicators for voltages from 0V to 1V.*

of minutes. Not only is this faster than manual optimization, but the process no longer requires manual labor—just the click of a button.

Future Work

Now that automatic rooftop end mirror rotation has been achieved, the signal-to-noise major has improved. However, the signal can be further improved by introducing automatic translational motion for the rooftop end mirror.

The microwaves make a standing wave because they are reflected by mirror. Therefore, the magnetic field maxima are stationary with a stationary mirror. However, with translational motion of the rooftop end mirror, the locations of the magnetic field maxima will change. If the position of the rooftop end mirror is changed to place the sample at a magnetic field maxima, the sample will have more opportunity for absorption. Therefore, a stronger signal will be produced. This future improvement, along with automatic rooftop end mirror rotation, will greatly improve the signal-to-noise ratio of EPR spectroscopy.

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