Research Paper

Author: Ruman Shaikh

Date: August 2022

Professor : Professor Doros Petasis

Summary

It is a device to measure the paramagnetic species it used for eliminating the radicals. The fundamental concepts of EPR are remarkably similar to those of the more common nuclear magnetic resonance spectroscopy, with the exception that EPR concentrates on the interaction of an external magnetic field with the unpaired electron in a molecule rather than the nuclei of individual atoms. EPR has been used to examine the kinetics, processes, and structures of paramagnetic species, and it has applications in biochemistry, polymer science, and geosciences, in addition to general chemistry and physics.

In this research paper we are going to examine three different bio-chemical samples using the EPR spectroscope,analyzing their paramagnetic resonance and finding the g-values for all of the bio-chemical samples allowing us to predict the structure of the compounds and help us understand the behaviour of the unpaired electrons .Futhermore,nuclear spins are calculated by the EPR Spectroscope which helps us predict the structure of hyperfine structures. The three samples which will be used are .In conclusion the EPR is a device of great importance as it allows us to gain the knowledge about the nuclear spins,the paramagnetic nature and the structure.

Background Information

The Epr and Zeeman Effect

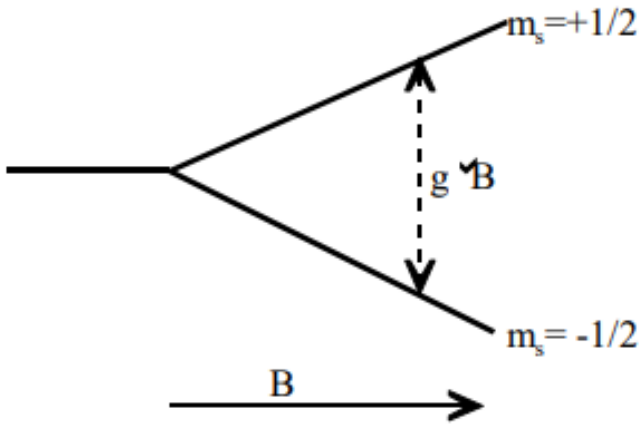
Hyperfine Lines

Hyper-Fine structure occur as a result of the magnetic interaction between electron spin(s) and the nuclear spin(i),due to the hyperfine structure there are several peaks or signals which split into several lines.

Epr

The reason for this is because EPR spectroscopy need unpaired electrons. Electron pairing is often energetically advantageous. As a result, only a limited percentage of pure chemicals exist. Consider this. Unpaired electrons, on the other hand, because electron pairing underpins the chemical bond, are related to reactivity as a result, EPR spectroscopy is a critical method.in order to comprehend radical reactions, electron transfer mechanisms, and transition metal catalysis, they are all connected to the 'unpaired electron's reactivity'. Spectroscopy has resolution limitations or cannot offer enough information for thorough analysis. Structure and dynamics characterisation.

Thus the electron has an intrinsic magnetic moment μe = -geS where g = 2.0023, e = eh/4%mc = 9.2741 × 10-24J T-1 (Bohr magneton), and S = ½



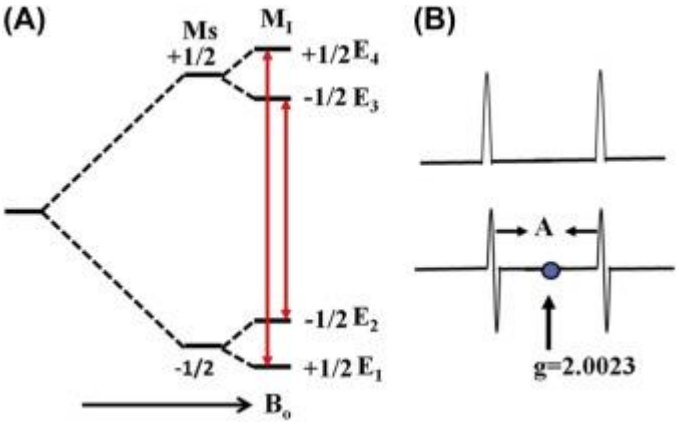
The Zeeman Effect of the First Order divides the ms state into two electrons that are unpaired in a Material that is paramagnetic. The resonance requirement is thus h = gB, but since An electron's magnetic moment is 2-3 orders of magnitude larger. larger than that of a magnetic nucleus, as measured by an ESR

spectrometer works at higher frequencies and with lower magnetic fields compared to an NMR spectrometer

Introduction

Hyper-fine Structure

Hyper-Fine structure occur as a result of the magnetic interaction between electron spin(s) and the nuclear spin(i),due to the hyperfine structure there are several peaks or signals which split into several lines .The splitting allows us to recognise the structure of the compounds.



To identify the following compounds

1. DPPH-dark-colored crystalline powder composed of stable free radical molecules
2. Myoglobin(Dry)(in water)(glycerol)- an iron- and oxygen-binding protein found in the cardiac and skeletal muscle tissue of vertebrates
3. Magnese(2) chloride -
   This inorganic chemical exists in the anhydrous form

More about hyper fine structure

Because the spin quantum number of even atomic number and even mass number nuclei is zero, no HFS is observed in their spectral lines. Other nuclei's spectra do show hyperfine structure. Nuclear spin may be calculated by monitoring HFS.

Isotope structure, or isotope shift, is a comparable effect to line splitting induced by mass differences (isotopes) of atoms in an element. These spectral lines are known as hyperfine structure and may be seen in elements with spin-zero

isotopes (even atomic and mass numbers). Isotope structure is seldom detected in the absence of real HFS.

Procedure

The device that is being used for this experiment is the Varian E-3 X-band EPR spectrometer along with a nitrogen cryo-stat.The spectrometers works from 8.5-12 giga hertz. The temperature range is from 85k till normal temperature. A microwave bypass arm is used to bias a diode detector for greater sensitivity. The signal from the machine is processed by the electronics allowing u to get the values. Then the spectra are digitalised using a scanner which can be printed out later on.

It is vital to note that during a low temperature EPR run, the temperature controller must be switched on after the coolant water is turned on. The main valve on the nitrogen tank, located behind the spectrometer, must then be opened. The temperature controller's tasks must be finished before liquid nitrogen may be injected into the storage. Once liquid nitrogen has been added to the storage, the sample being examined must be gradually lowered into a second storage containing liquid nitrogen. It is critical to go slowly so that the EPR tube does not break. Once the sample has cooled, it may be put in the cavity. Then, the same processes as in a normal temperature EPR .

Experiment

DPPH:For DPPH,a miniscule amount is taken and placed into a clean EPR tube and sample of DPPH is placed into a grease spot.EPR measurement are proceeded at the temperature of 298 kelvin with a Varian E-3 X band spectrometer.

Myoglobin(Three Samples):

1. Dry Myoglobin-A sample of the myoglobin is put in the Epr spectroscope at room temperature.
2. Myoglobin in water-with water as a solvent is to be taken in an EPR tube.
3. Myoglobin in glycerol-A sample of myoglobin in glycerol as a solvent was inserted in an EPR tube, and measurements were taken at 110K.

Magnesium chloride : $MnCl_2$ was placed in an EPR tube, and measurements were obtained at 104K.

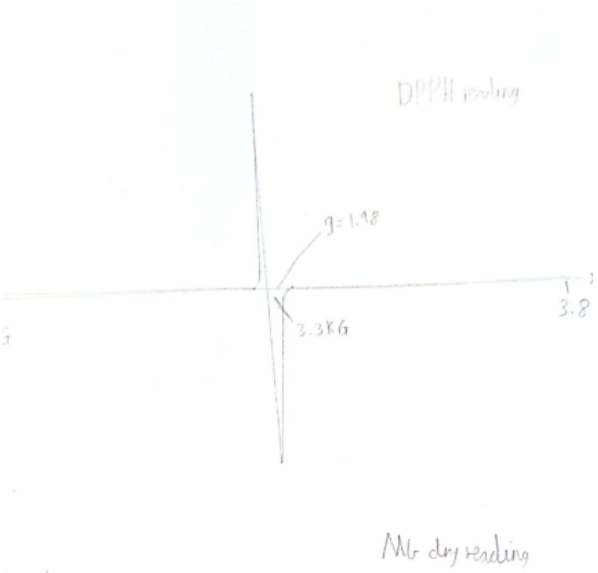
The first step in running the Varian E-3 Spectrometer is to switch on the cooling water situated beneath the fume hood. This prevents the magnet from overheating. The spectrometer and universal counter should be turned on while doing a room temperature scan. To obtain an accurate reading, the frequency channel must be adjusted to channel three. The oscilloscope's intensity must then be adjusted all the way to the right. When a signal is detected, the sample is put into the cavity. To tune, adjust the mode knob at this stage. The oscilloscope will then reveal a dip. The horizontal location of the dip is then adjusted till it lines up with the black line on the oscilloscope. The mode knob can be twisted to operate once the dip is in the right position.

The recorder switch can be switched on at this stage. The magnetic field mid-range and scan range are then configured, and the scan button is pushed to the right to

start a room temperature EPR run. It is vital to note that while doing a low temperature EPR run, the temperature controller must be switched on after the coolant water is turned on. The primary valve on the nitrogen tank, which is positioned behind the spectrometer, must then be opened. Before liquid nitrogen can be introduced into the dewar, the temperature controller's activities must be performed. Once liquid nitrogen has been added to the dewar, the sample being examined must be progressively lowered into a second dewar containing liquid nitrogen. It is critical to move slowly so that the EPR tube does not break. After cooling, the sample can be inserted in the cavity. The same actions can then be taken as in a normal temperature condition.

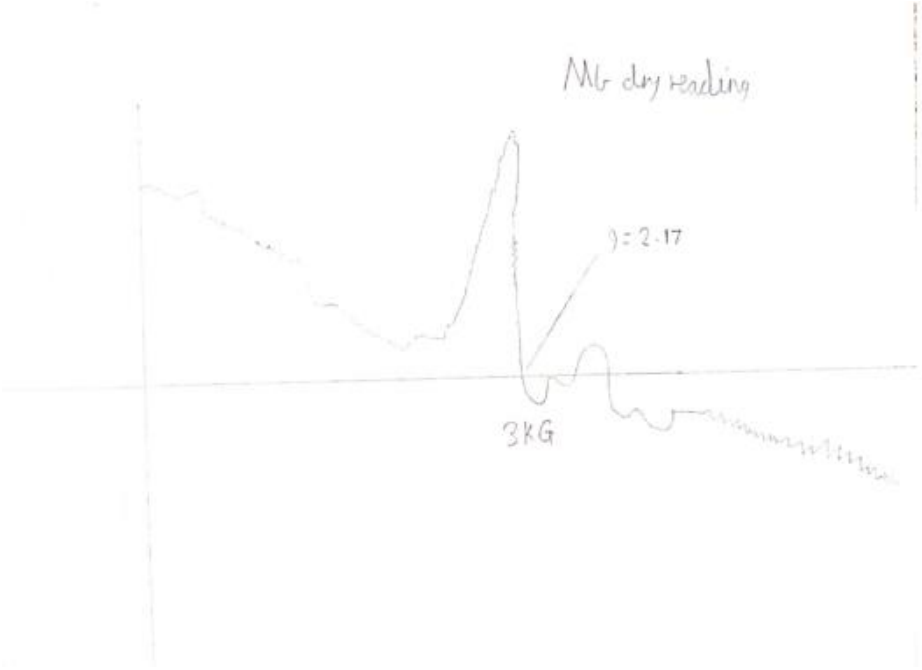
Conclusions

DPPH-using the formula we arrive on the value of g to be 2.01 when measured at a temperature of 298k. DPPH sample is a free radical as the theoretical value of g is 2.00 as the value is near this value there might be an error as error percentage is 1 percent.

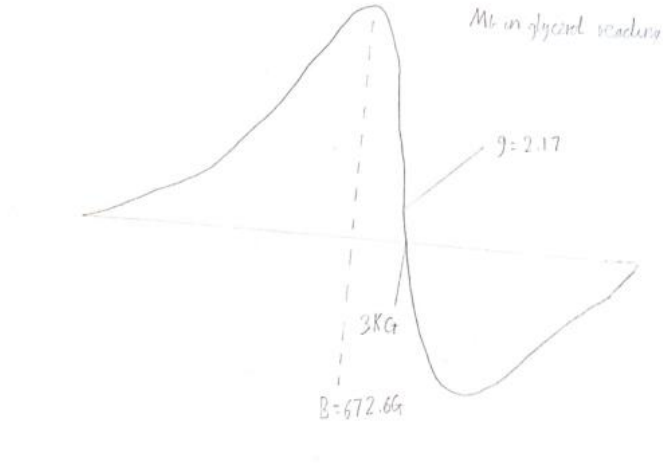


Myoglobin-Out of the three samples of myoglobin two of them show the same results of the g-factor being 1.9 proving that myoglobin isnt a free radical. As for

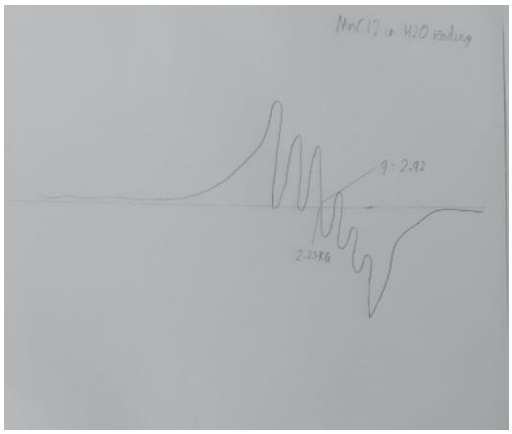
the Myoglobin in glycerol was 1.9 as well therefore not a free radical.



However, when the g-value of Mb in H2 O is calculated, it comes out to be 2.2 The variation in g-value between the dry and wet myoglobin samples is attributable to the difference in concentration. properties of the two solvents.



Mncl2 H20-the g-value of mncl2 is 2.5 therefore, being a free radical in the state of bounded electron of Mn2+.



Table

DPPH	**2.01**
DRY MYOGLOBIN	1.9
MYGLOBIN IN GLYCEROL	1.9
MYGLOBIN IN WATER	2.2
MNCL2	2.5

REFRENCES:

W.R. Hagen, “Practical Approaches to Biological Inorganic Chemistry”, Second Edition, 2020.

.D.Petasis , EPR handout 1,2, “Introduction to College Level Research in Physics”, LS 102, Allegheny College

John. A. Weil, James R. Bolton, Electron Paramagnetic Resonance Elementary Theory and Practical Applications, Illinois, Wiley,2007.

Physical Chemistry IV Part 2: Electron Paramagnetic Resonance Gunnar Jeschke