

Instrumentation Control for Pulse EPR Spectroscopy

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Abstract

A collaborative project is described involving the Milwaukee School of Engineering and the Medical College of Wisconsin, and the National Institutes for Health. The purpose of this project is to design and implement a computer-based instrumentation control system for an electron paramagnetic resonance (EPR) spectrometer utilizing pulse excitation. Background information on the application of EPR spectroscopy in the context of this project is provided. The design of the control system is presented in detail with particular emphasis on the event-driven program structure.

Introduction

This paper describes a capstone electrical engineering technology (EET) senior project accomplished as a joint effort between the Medical College of Wisconsin (MCW) and the Milwaukee School of Engineering. The project was funded by grants from the National Institutes of Health. The two-quarter project consists of an independent study to form a background into the methods and hardware involved in EPR systems, with a second quarter devoted to software development of the actual control system.

The goal of the project is to provide control capability by way of a LabVIEW virtual instrument utilizing GPIB connectivity. The software interface adjusts the timing features of the instruments in the Q-Band Pulse EPR system used at MCW.

An EPR spectrometer that employs pulse excitation can be either of the commercially available variety or can be assembled from off-the-shelf microwave and electronic components. With the research work at MCW being more instrumentation-oriented, the discussion focuses on an EPR system of the latter type. Before addressing the development of the control software, a primer on the methodology and hardware of EPR systems is presented.

EPR Background

Scientific research uses EPR in many ways to uncover answers about matter and behavioral effects in matter at the atomic and quantum levels. Various scientific disciplines utilize EPR as an investigative tool:

- Physics – susceptibility, semiconductors, quantum dots, defect centers
- Chemistry – et-reaction kinetics, organo-metallic, catalysis, molecular magnets
- Ionization radiation – alanin radiation dosimetry, radiation damage, irradiated food
- Material research – polymers, glasses, superconductors, corrosion, dating
- Biology – enzyme reaction, et-reaction, folding & dynamics, metal centers, free-radicals, and spin-labeling, including nitroxide spin labels attached to cysteins or nucleic acids

There are several implementations of EPR technology:

- CW (continuous-wave) EPR
- Pulse EPR
- ENDOR (electron double resonance) EPR
- ELDOR (electron-electron double resonance) EPR
- ESEEM (electron spin-echo envelope modulation) EPR
- Multi-quantum EPR



Figure 1 – Developmental EPR System

An EPR spectrometer (Fig. 1) in general terms is a device that analyzes the amount of energy absorbed or given off by a particular sample when it is irradiated by a sweeping of an electromagnetic energy field (or band of frequencies). The resulting spectrum reveals various aspects of the sample's molecular structure and its behavior. For example, X-Ray spectroscopy uses very short electromagnetic waves to cause electrons to shift quantum levels. In doing so, the quantum shift causes absorption or release of energy depending upon the angle of incidence of the crystalline structure. The intensity of this energy returned to the detector will yield the spectrum. EPR spectroscopy is similar, although the ionizing capability of microwave electromagnetic energy is much less than that of X-rays. And, instead of looking at crystalline structures, the behavior of unpaired electrons in the molecular structure of a material is examined.

Two key aspects are central to EPR operation. First, since electrons are spinning spheres of charge, they have a magnetic moment. This magnetic moment is similar in behavior to a typical

bar magnet. Under the influence of a magnetic field, the direction of the electron's moment can be altered. This behavior is founded upon a concept called the Zeeman Effect, which states that an external magnetic field will exert a torque on a magnetic dipole. Secondly, it is necessary to have unpaired electrons available in the molecular structure of the substance under test. An unpaired electron is one which has no partner in an unfilled outer shell. The unpaired electrons are needed because the spins of paired electrons in completed shells tend to cancel each other out. Since the spinning effect is what yields the spectrum, it is important to have this electron alone to manipulate and measure its response.

In EPR spectroscopy, the frequencies of microwave radiation are commonly 3-180 GHz. At these frequencies, there is not enough energy to cause a quantum shift, but an important phenomenon known as precession occurs with the electron itself. Precession happens when the magnet in the spectrometer system causes magnetic lines of

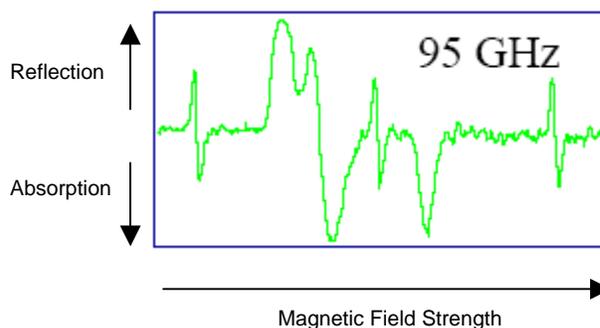
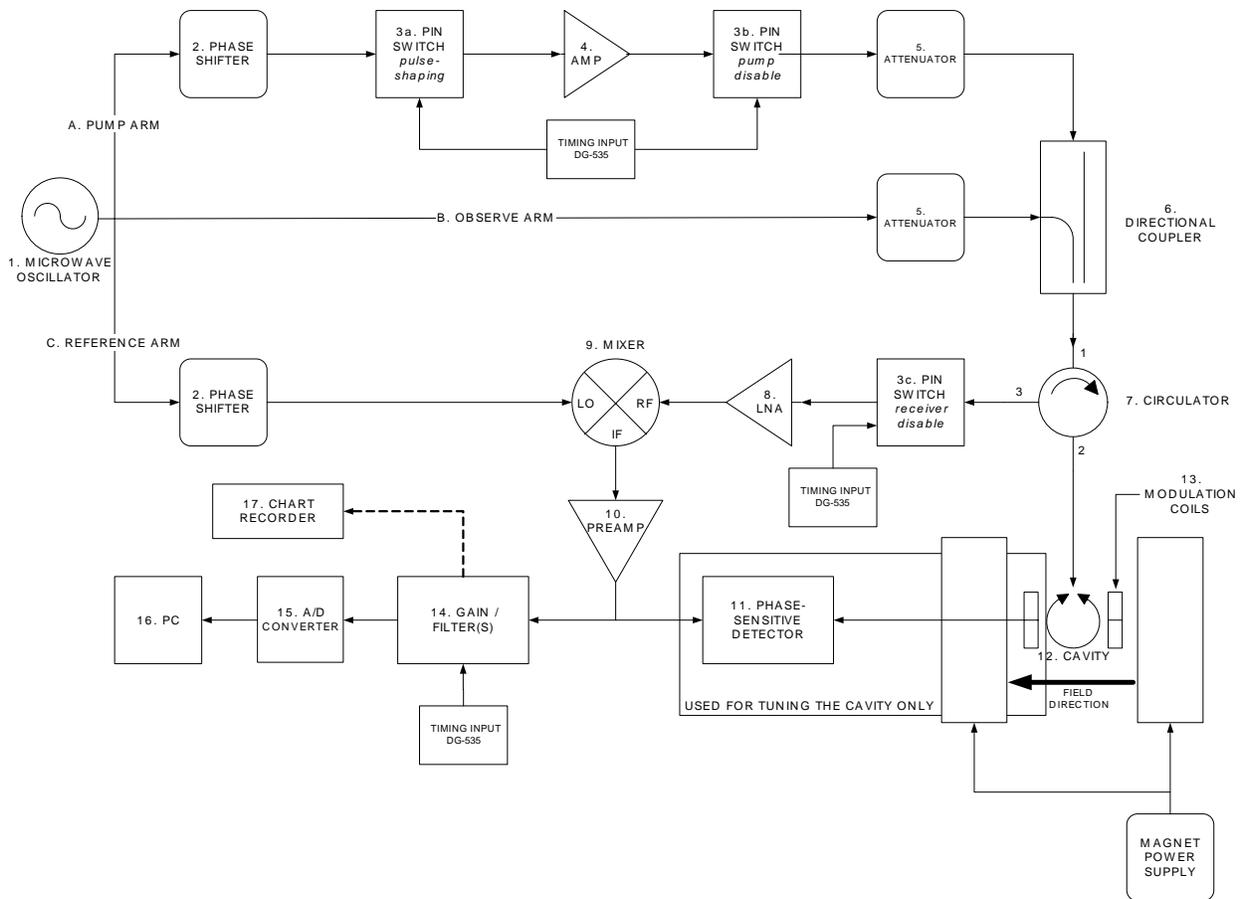


Figure 2 – Typical EPR sample response

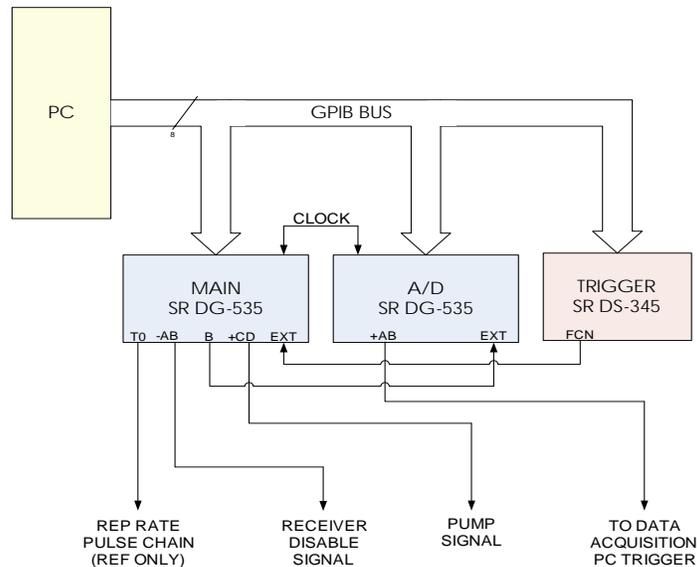
force across the resonator cavity, which causes the electrons to precess, or wobble around their axis. The greater the magnetic field, the more pronounced the precession. The microwave energy field is introduced into the cavity perpendicular to this strong magnetic field. This causes a 'tipping' of the cone of precession between 90° and 0° . It is between these two angles that EPR information is derived, as more or less of the microwave energy is absorbed by tipping the cone of precession. When absorption occurs, a dip is noted in the spectrum, and conversely, when dispersion occurs (reflection of microwave energy), there is a rise in the spectrum (Fig. 2). These reflections, due to the absorption or dispersion of energy, are part of the EPR spectrum; which give the scientist insight into the sample's molecular structure and behavior.

The pulse EPR methodology allows a very short burst of microwave energy to the cavity's sample (Fig. 3). The power level, duration, and repetition rate of these bursts are determined by the experimenter, based on the sample's properties and the kind of EPR information desired. For example, a long pulse width will fully irradiate the sample and cause all of the moments of the sample's molecular structure to be affected. In a short pulse setup, isolation of areas that respond more quickly to the microwave's influence can be studied. The repetition rate is set to perform multiple analyzations and promote averaging of the sample's response.



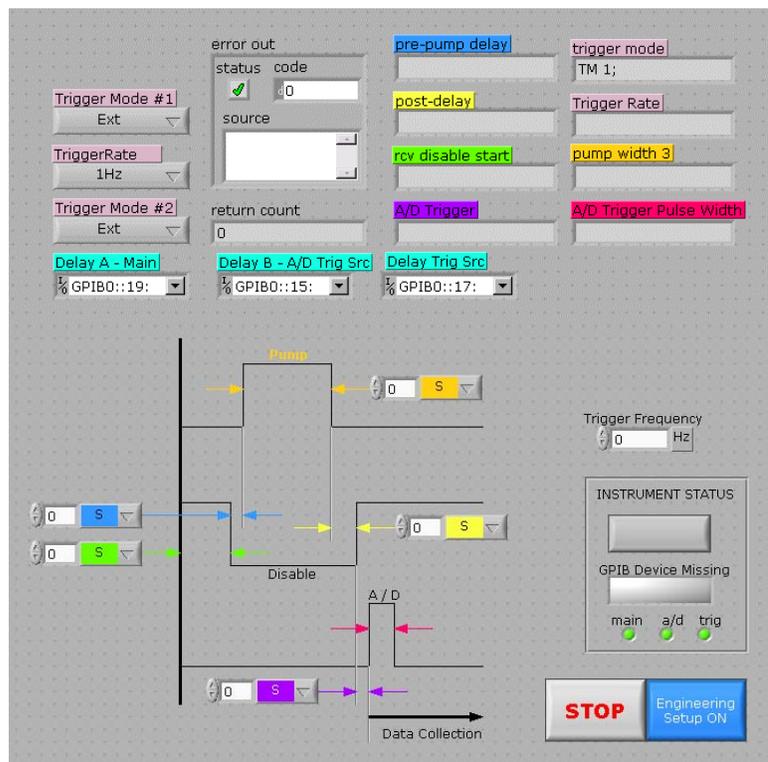
The timing sequences are applied to four key areas on the microwave circuit. These signals are provided by two Stanford Research (SR) DG-535 pulse/delay generators (Fig. 4). The trigger frequency of the DG-535's is controlled by an SR DS-345 function generator. All necessary features of these instruments will be under software control. Referring to the microwave circuit of figure 3, the three pin switches 3a, 3b, and 3c, along with the A/D converter (block 15) are the areas where timing signals from the SR instruments will be sent. These are the areas to which software control of the timing sequences will be applied. With these specific areas known, a control system can then be designed to accommodate adjustments and setup remotely. Hardware implementation of the SR instruments is shown in the block diagram of figure 5.





Software Implementation

A LabVIEW virtual instrument was developed to provide the scientist, who is the end user, with a more user-friendly and intuitive interface with which to setup experiments on the EPR system. It is important to note that before the LabVIEW interface was developed, input of the proper timing signals would have to be programmed from the less intuitive front panels of the two DG-535's (Fig 4). The front panel that the user sees is shown in figure 6. However, the top part is only accessible to the engineer for GPIB setup and troubleshooting tasks, and will not normally be seen by the user.



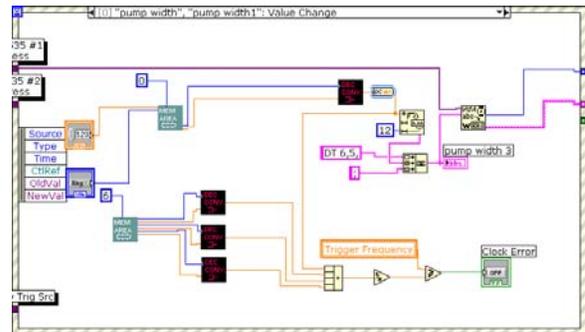
The four main objectives of the software project included:

- Modularity of code blocks
- Restricted access to front panel controls
- Graphical and user-friendly display elements
- Error-handling routines and checks

Secondary objectives, which were added during the design process included:

- Control of multiple instruments on the GPIB bus
- Adjustment provision for A/D trigger width

The foundation of the control software is based on an event-driven structure, which is available in LabVIEW versions 7.0 and later. The event-driven structure centers on the ability of the software to refrain from using computer resources (unlike polling) unless a parameter in the front panel is changed. This greatly frees up system resources that can be allocated to other tasks. In the case of the pulse system at MCW, these freed resources are used for data acquisition duties. The basic event structure is shown in figure 7, and allows front panel controls to be assigned to trigger an event or events when changed or updated. When an event is triggered, the program engages, updating the current value in memory and sending the correct instructions to the instrument on the bus. Sending the instructions is accomplished through use of the VISA read/write functions, assembling the proper command syntax of the instrument combined with front panel input.



In addition to the event-driven structure, a memory storage mechanism called a VIG (virtual instrument global) is employed. A VIG is a sub-vi called whenever parameters are changed via the front panel. It utilizes several shift registers, one for each parameter that needs to be stored and recalled (Fig. 8). In this program, there are three possibilities for calling the VIG: a "set all" operation, a "get all" operation, and setting individual parameters. The "set all" and "get all" are used in conjunction with instrument initialization, which allows setting the SR generators with default parameters when the program is started. This also ensures proper operation and protection of the EPR system from signals that would cause damage to the microwave components.

