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Autobiography of James S. Hyde

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Abstract

The papers, book chapters, reviews, and patents by James S. Hyde in the bibliography of this document have been separated into EPR and MRI sections, and within each section by topics. Within each topic, publications are listed chronologically. A brief summary is provided for each patent listed. A few publications and patents that do not fit this schema have been omitted. This list of publications is preceded by a scientific autobiography that focuses on selected topics that are judged to have been of most scientific importance. References to many of the publications and patents in the bibliography are made in the autobiography.

1. FAMILY AND SCHOOLING

I was born May 20, 1932 in Mitchell, South Dakota into an academic family. My father, Melvin Watson Hyde, held a PhD degree from Columbia University in New York City, and my mother, née Claudia Marie Bray, was a pianist with a master's degree from the Cincinnati Conservatory of Music. My father held several university positions in the Midwest, ending his career with a distinguished tenure as president of the University of Evansville, in Evansville, Indiana. I matriculated into the Massachusetts Institute of Technology (MIT) in Cambridge, Massachusetts, in 1950, graduating in 1954 with a degree in physics. I then entered the graduate school at MIT in physics with specialization in solid state physics and received a PhD degree in 1959. I was employed at Varian Associates in Palo Alto, California for 16 years, became professor of biophysics in radiology at the Medical College of Wisconsin (MCW) in 1976, and became professor emeritus in January 2016. My wife (Karen) and I live in Wisconsin. Our three children are married, each with two of their own children.

I spent an interesting summer in 1953 at the DuPont Experimental Station in Wilmington, Delaware, working as an intern on the physics of clothing comfort. My earliest scientific success was my undergraduate thesis written in 1954, "Quasi-static Ferroelectric Hysteresis Loops of Barium Titanate." Prof. Arthur R. von Hippel served as thesis advisor. I designed and built the apparatus and carried out the experiments.

The summers of 1954, '55, and '56 were spent in Los Alamos, New Mexico working for the Atomic Energy Commission in the Test Division. I found myself calculating bomb yields in 1954, using methodology that had been developed by Hans A. Bethe, searching for possible loss of efficiency of the photographic process associated with the mobility of "holes" in AgCl in 1955, and participating in experiments in the South Pacific in conjunction with the first hydrogen bomb air-drop, which I witnessed, in 1956 (Operation Redwing).

My PhD research was carried out in the Laboratory for Insulation Research directed by Prof. von Hippel, with supervision by Assistant Prof. Perry A. Miles. This was an interdisciplinary laboratory based in the Department of Electrical Engineering with graduate students from a number of departments. I was a student in the Department of Physics. My dissertation title was “Magnetic Resonance, Relaxation and Rapid Passage Phenomena in LiF F Centers.” It resulted in two papers in the *Physical Review* [4, 84]. Paper [4] was a theoretical calculation. I recognized a flaw in the Bloch equations: They did not properly account for the off-resonance transition probability that depended on the amplitude of the microwave field in a continuous wave (CW) experiment.

The electron paramagnetic resonance (EPR) spectrometer that I designed and built was at K-band (24 GHz). It employed a transmission cavity with a bucking arm in the cavity output to reduce the transmitted microwave level. It incorporated an alternating current (AC) Pound circuit for the automatic frequency control (AFC). I was, for the most part, on my own in development of this system, although there was good engineering support for the construction of the lock-in detector system. My experience with the AFC system surfaced recently, reported in the appendix of a paper on a W-band EPR system [215]. This AFC system uses contemporary digital technology but, nevertheless, has conceptual similarity to the ideas of Pound.

Interest in passage phenomena remained high throughout my career. It led directly to the discovery of saturation transfer (ST) EPR, which extended the range of motion that could be studied by spin labeling to include rotational diffusion of macromolecules [Topic XI]. The papers by Prof. D. D. Thomas and his students apply this methodology to the muscle-protein system [164, 167, 168, 171, 172]. More recently, my graduate student, Aaron W. Kittell [76; see also, Topic XIV], used the technique of non-adiabatic rapid sweep (NARS) to study the early evidence of adiabaticity. Kittell’s work describes the gradual transition from stationary CW EPR to the onset of free induction decay (FID) and the appearance of passage phenomena.

2. VARIAN ASSOCIATES

Post Sputnik, jobs were plentiful. I skipped the postdoctoral phase, going directly to the quasi-academic Analytical Instruments Division of Varian Associates in 1959. Nuclear magnetic resonance (NMR) and EPR were in the same group. One might say that my postdoctoral training occurred during lunches with some very strong NMR scientists, including Richard R. Ernst, Wes Anderson, and Ray Freeman. Later in my career, I became intensely involved with magnetic resonance imaging (MRI). It seems likely that this early immersion in an NMR environment had been quite helpful. Felix Bloch and Harden McConnell were consultants. I wrote 60 papers and was awarded 13 patents during the 16 years that I was at Varian [Topic I].

A. H. Maki found himself with some time between two faculty positions, and he and I decided to make an all-out push to detect electron nuclear double resonance (ENDOR) in liquids. He picked the system (galvinoxyl), and I assembled a kilowatt of radio frequency power. I was forced to order another hundred reprint copies to meet the requests that we

received for the paper describing our work [31; see also, Topic VIII.IV]. At that point, ENDOR was the most exciting science that I had ever done. One of the great pleasures of being employed by Varian is that I was able to support a postdoctoral fellow. My first postdoctoral fellow, Gunter Rist, was from ETH Zurich, in Switzerland. He and I created the foundations of ENDOR in powders and frozen solutions [Topic VIII.III]. The idea of selection of orientation occurred to me, enabling us to obtain well-resolved NMR spectra by EPR observation at “turning points.” Anders Ehrenberg from Stockholm, Sweden introduced me to the “Old Yellow Enzyme” [96]. We carried out a successful ENDOR experiment on this flavo-protein, the first ENDOR experiment on a protein. Rist’s expertise on copper then led, with more Swedish connections, to the first ENDOR experiment on a metalloprotein [99]. I traveled to Europe for the first time to present an invited lecture on ENDOR in Cirencester, United Kingdom.

I developed a rectangular bimodal resonant cavity system in which portions of the two modes overlapped in the sample regions, and other portions supported only one of the modes [269]. Insertion of plastic or metal into these single-mode regions permitted tuning of the two resonant frequencies. This structure enabled three new experiments—electron electron double resonance (ELDOR) [32; see also, Topic IX], pulse saturation recovery [144–146; see also, Topic X], and convenient tuning between H_1 parallel and perpendicular to the static magnetic field for precision characterization of forbidden transitions.

In addition to Varian postdoctoral fellows, there were several fellows with me who had their own support. A number of them have had distinguished scientific careers. We ran workshops on a regular basis, since magnetic resonance techniques were new and few investigators had formal training in this field. Short visits from well-established investigators were common. I was exposed to a very wide range of applications of EPR spectroscopy, and I met a number of investigators who, in later years, were important to the development of the National Biomedical EPR Center at MCW.

In the 16 years that I worked at Varian, my papers brought scientific recognition and invitations to lecture at international meetings. I became active in the series of conferences known as Magnetic Resonance in Biological Systems, traveling widely. I also traveled for business reasons. A notable trip was to Moscow for what may have been the first commercial exhibition in the Soviet Union by an American company. The exhibition was enabled on the Soviet side by Vladislav V. Voevodsky, a noted physical chemist whom I had met in Palo Alto. I gave a lecture for the group of Prof. L. A. Blumenfeld, with whom I struck up a decades-long friendship. My lecture was on my ELDOR experiments. At the close, I was introduced to Victor Benderski, who told me about his current work on ELDOR using a completely different methodology. I consider Benderski also to be my friend. We had an E3 X-band spectrometer working at the exhibition. A student from Poland used it nightly for the week of the exhibition; I understand this enabled him to complete his master’s thesis. This student was Wojciech Froncisz, with whom I later authored about 100 papers. Another Polish student who is prominent in my memory is Tadeusz Sarna. Because of his excellent English, he was constantly with me as an interpreter. Sarna also became a lifelong scientific colleague. I made a number of visits to the former Soviet Union, including two trips to Kazan, the home of the discoverer of EPR Spectroscopy, Yevgeny Zavoisky. I

recall seeing one of his instruments on display in a museum. I met him, as well as his colleague S. A. Altshuler, during a meeting to celebrate the 25th anniversary of the discovery of EPR.

On one of my trips to Novosibirsk in Siberia, I met Anatoly G. Semenov, who was, I understand, the lead engineer in the development of the commercial EPR 2 spectrometer, a version of which was distributed in the Soviet Union. He asked me a question: Why did Varian make the cross-section in the X-Z plane of the multipurpose TE₁₀₂ cavity rectangular rather than square? I immediately understood his point: The plane of zero electric field in which a flat cell with water sample can be located could be larger, thereby improving the concentration sensitivity (i.e., unlimited sample but fixed concentration of paramagnetic radicals). I had nothing to say. We just had not thought of that possibility. Flying home, I thought more about his question, and I had an idea: If I made the cross-section square but also rounded off the corners so that the square morphed into a circle, it would be even better. That microwave mode is actually known: TM₁₁₀. I built it, turned it into a successful commercial product, and obtained a U.S. Patent on the idea [274]. It was the first time the TM₁₁₀ microwave mode had been used for EPR spectroscopy. And it remains the all-time winner for concentration sensitivity. Such is the benefit of travel.

I became increasingly interested in spectra from nitroxide radical spin systems that were undergoing very slow rotational diffusion. I called it the “very slow tumbling domain” [Topic VII]. In this domain, motional narrowing is no longer apparent, and the ordinary CW spectra appear to be in the rigid limit. However, spectral diffusion continues to occur on the time scale of the electron spin-lattice relaxation time, resulting in effects that can be detected at higher incident microwave power, including passage effects. I continue to probe this domain, which I consider to be one of my scientific discoveries, even as a professor emeritus.

I also became interested in relaxation between spins that were contributing to EPR intensity in various spectral regions. These mechanisms include nitrogen nuclear relaxation in spin labels and Heisenberg exchange between diffusing spin labels [Topic IX]. The foundation was laid for an extensive body of work in collaboration with my colleague J. B. Feix at MCW based on Heisenberg exchange between ¹⁴N and ¹⁵N spin-labeled species [36, 135–137].

My experiments were increasingly pulling me toward biological applications. I was awarded a short sabbatical leave and spent three months in 1973 with my family in Sweden, working in Prof. A. Ehrenberg’s very active laboratory at the Karolinska Institute in Stockholm. An interesting paper emerged, which was related to the physics of the very slow tumbling domain [20]. I was starting to think of myself as a biophysicist. Back in California, I found the environment at Varian to be trending more toward the commercial side, and less academic. Two of the NMR scientists, Ernst and Freeman, had left.

I wanted the Varian commercial EPR business to do well. That segment was headed by a project manager who was responsible for running the EPR segment profitably. My most successful contribution was the introduction of a new microwave frequency for EPR: 35

GHz or “Q-band.” We sold about 100 Q-band bridges, of course with larger magnets. This was the first Varian bridge with a reference arm. I extended the wire-wound concept for a TE_{011} cavity to Q-band. I made the decision that the tunable range of the klystron was too small, and therefore the sample resonator had to be tunable. I helped the magnet designers develop “ring” shimmed tips that could be inserted into the magnet gap to produce a homogeneous field at 12,000 Gauss. The system was engineered first in the “V-line” series, and later in the “E-line” series. Some of these systems are in use to this day. An updated version of the bridge with a low-noise microwave amplifier is described in the literature [213]. I felt that I was making a meaningful contribution to science through my commercial endeavors.

From the earliest days, we were attempting to develop a market for EPR spectrometers. We concluded that this goal required us to engage in the teaching of EPR spectroscopy. Our series of workshops led to the publication of an early book, *NMR and EPR Spectroscopy*, by the NMR-EPR Staff of Varian Associates, Pergamon, New York, 1960. The series of scientific advertisements, *NMR at Work*, was supplemented by a second series, *EPR at Work*. They were each one page and published, for example, in *Physics Today* or *Chemical Engineering News*. There was a brisk claim about a feature of an instrument, followed by a novel scientific story with references. I wrote about half of the *EPR at Work* series. They have now been published as a set, see Topic I [3], making them widely available and still of some interest.

I also wrote a few longer articles for the *Technical Information Bulletin*. In one of them, I introduced a classification system for types of EPR samples. Three questions had to be answered “yes” or “no”: Was the sample limited in availability? Was it saturable with the available microwave power? Did it exhibit dielectric loss? My point with these questions was that a range of microwave resonators could be developed for optimum sensitivity across a range of samples. This has become a lifelong perspective, impacting my most recent work. This article has also been reproduced in Topic I [3].

Some years later, after I had moved to MCW and Varian had withdrawn from the EPR market, I came to realize that technology and lore were in danger of being lost forever. I took advantage of an invitation to write a chapter in the two-volume set edited by T. K. Ishii, Topic I [1], in which I tried to preserve some of this Varian knowledge.

3. BIOPHYSICS AT MCW

The National Center for Research Resources (NCRR) was a funding agency within the National Institutes of Health (NIH) that had a mission to fund “research resources.” These were defined as Biomedical Research Centers, each of which focused on a particular technology or capability that was not sufficiently large to attract commercial companies but nevertheless was important to biomedical research. These research resources often can be characterized as having highly specialized technologists with expertise that is not widely available. A research resource is expected to have both the engineering capability to advance a field and the biological expertise to advance the applications of the technology. The

equipment should be freely available to NIH-funded investigators. Often the leadership of a research resource was dual, with a technologist and a biomedical specialist.

Harold M. Swartz, MD, PhD, at MCW thought that he and I could serve as co-leaders in an initiative to establish the first EPR research resource. For me, it was a perfect fit. I accepted an appointment as full professor of radiology beginning in 1975.

We were successful in our first application; Swartz was principal investigator. Three years later, he accepted a position at the University Illinois, and I became principal investigator. The National Biomedical EPR Center, or the “Center,” was funded for 42 years, surviving an NIH traumatic event when the NCRR was closed and the National Institute of Biomedical Imaging and Bioengineering (NIBIB) was established by an Act of Congress. Within MCW, we were initially a section of the Department of Radiology, and I was the section chief. We later became the Biophysics Research Institute, and I was the director, reporting to the dean of the Medical School. I stepped down as institute director in 1998. Appointment of Balaraman Kalyanaraman to replace me led to the creation of the Department of Biophysics, with Dr. Kalyanaraman as the department chair. From an organizational perspective, the Center, which I continued to direct, was a center within the Department of Biophysics.

Shortly after I came to MCW, Swartz began the lengthy process of establishing an accredited training program in biophysics leading to the PhD degree. Over the years, we had many young research colleagues and granted about 100 PhD degrees. In 2009, I was awarded a personal chair: The James S. Hyde Professor in Biophysics. MCW awarded me further internal recognition for my contributions to the college: The Distinguished Service Award in 2008 and The T. Michael Bolger Award in 2012. I became professor emeritus in January 2016 by action of the board of trustees.

4. THE NATIONAL BIOMEDICAL EPR CENTER

Our first major task was to establish an EPR engineering capability. I was not successful in bringing along any of the engineers with whom I had worked in California. Eventually, I was able to secure the agreement of ETH Zurich for Jorg E. Forrer to come to Milwaukee, Wisconsin for two or three years to help me build an EPR engineering capability. He tapped into the pool of graduates of the Milwaukee School of Engineering (MSOE). Two engineers, Joseph J. Ratke and Theodore G. Camenisch, were identified and trained, and two MSOE professors, Robert A. Strangeway, PhD, and Richard R. Mett, PhD, spent summers and additional time depending on funds available at the Center.

Prof. S. Lukiewicz from the Jagiellonian University in Krakow, Poland, and Swartz and I on behalf of MCW made a formal exchange agreement in the middle of the Cold War. Amazingly, my old friends from Moscow, Fronecisz and Sarna, were in his group. They had built an experienced core of EPR engineers. We began to have exchange visits involving not only the Polish EPR scientists, but also the Polish EPR engineers.

The NIH provided us with excellent equipment funding, which enabled us to establish an impressive microwave engineering facility. A machine shop was established, with many of the machines under numeric control.

At the same time that I was building the engineering capability of the Center, biomedical research scientists trained in EPR were being recruited as members of the Biophysics faculty. A model was established: An EPR technology development core surrounded by a circle of experienced EPR users. The individual networks of these users would attract users of the Center from other institutions. For many years, about 50 scientific papers annually cited the Center's grant number.

5. EPR RESEARCH

5.1. Multi-frequency EPR and the Discovery of the Loop-Gap Resonator

On Dr. Froncisz's first visit to the Center, I asked him to construct an S-band bridge. I foresaw that multi-frequency EPR could be a perfect fit to the mission of the Research Resource Program. Few places could afford to develop a wide array of microwave frequencies for EPR spectroscopy, but there were many situations where a specific frequency could be valuable to the research of a visiting scientist. I assisted in the development of a cavity for the 2–4 GHz octave bandwidth bridge that was constructed. It was scaled after the Varian multi-frequency EPR cavity, with an interesting extra feature. It oscillated in the rectangular TE_{104} mode at 4 GHz and TE_{102} mode at 2 GHz. Thus, the same sample could be examined at two microwave frequencies in the same scan session. The first experiment was exciting: Hyperfine structure resolved by 2–4 GHz EPR of cytochrome *c* oxidase, by W. Froncisz, C. P. Scholes, J. S. Hyde, Y.-H. Wei, T. E. King, R. W. Shaw, and H. Beinert, published in the *Journal of Biological Chemistry* in 1979 [191]. Spectral resolution was spectacular, apparently because of reduced strain broadening. Strain broadening in Cu^{2+} was discovered and analyzed the next year [192]. A review was published very early [184]. The spectrum of cytochrome *c* oxidase was difficult to interpret. Eventually, it led to the discovery of the mixed valence copper site with one unpaired electron distributed over two copper atoms. The cavity resonator worked well enough, but the amount of sample required was far too large for it to be widely practical. A new approach to resonator design was clearly needed.

The loop-gap resonator (LGR) was reported in 1982 [33]. This paper is my most-widely cited EPR publication. We went on to develop microwave bridges in octave bandwidths from 0.5–1 GHz, 1–2 GHz, 2–4 GHz, and 4–8 GHz. We made a small production run of 10 LGRs with variable match and robust sample support, each with the same diameter sample loop but with a different gap. We soon had LGR resonators at microwave frequencies from 0.5–10 GHz. Q-band was added to the list of LGR frequencies in 1986 [37], and W-band in 2007 [50]. The LGR was an enabling structure for multi-frequency EPR at low microwave frequencies and allowed novel experiments at higher frequencies because of the good sensitivity for extremely small samples. (Topic IV, EPR Resonators, lists more than 20 papers on LGRs.) The LGR also led to our extensive research in MRI, as discussed subsequently in this autobiography.

Paper [43], published in 1996, was the first to make extensive use of the finite element modeling of Maxwell's equations. We have extensively used computer modeling of electromagnetic fields since that time, as written in many papers by J. W. Sidabras, R. R. Mett, and J. S. Hyde in all permutations of the names. Mett became adept at developing

analytical solutions to resonator problems over a continuous range of input parameters that could be checked by finite-element modeling at discrete parameter values.

5.2. Saturation Recovery EPR at MCW

I wrote two papers on saturation recovery while at Varian that were published in the *Review of Scientific Instruments*, one with Martin Huisjen [145] and one with Paul W. Percival [146]. The instrument used a single channel boxcar for signal acquisition, making it very slow. The bottleneck was data transfer and accumulation. Engineer Forrer already had a plan for speeding the method up when he came to Milwaukee, Wisconsin to help establish the engineering capability at the Center. With eight parallel channels, time became available for transfer and accumulation. The first report of success with the new instrument was in 1980 [147]. A series of papers was published in the Proceedings of the National Academy announcing various capabilities of the new instrument.

The first of these, by C. A. Popp and J. S. Hyde [133], appeared in 1982. It provided the first saturation recovery data in a spin-labeled model membrane system (dimyristoylphosphatidylcholine [DMPC]). Bimolecular collision rates were measured at much lower concentrations than previously used for ordinary EPR in spin-labeled membranes to study diffusion. A bimodal cavity resonator was used as described in papers [145, 146].

Popp's experiments used the TPX gas exchange method to remove oxygen from samples [240]. It occurred to A. Kusumi, W. K. Subczynski, and me as reported in another *Proceedings of the National Academy of Sciences* (PNAS) paper [246] to use the gas exchange method to introduce oxygen to the sample in a controlled manner with the goal of studying oxygen transport in membranes. They introduced the oxygen transport parameter as a measure of bimolecular collisions of oxygen with a stearic acid spin-label moiety at a defined depth within the model membrane system. The work came together in a methodology to permit calculation of the oxygen permeability of a membrane. No competing methodology exists for this measurement, and it remains a key capability in ongoing studies of Subczynski. The year was still 1982, the year the LGR was discovered. It took some time to incorporate it into the saturation recovery system.

In a novel application of the methodology of saturation recovery, P. Fajer, a user of the Center's facilities from the David D. Thomas laboratory at the University of Minnesota, observed recovery of saturation because of rotational diffusion of a spin-labeled macromolecule. This 1986 paper [172] refers to the LGR used in the saturation recovery apparatus. It was a major advance.

My student J. J. Yin, along with me and Marta Pasenkiewicz-Gierula, published another PNAS paper on saturation recovery in 1987 [149]. Bimolecular collisions between ^{14}N - and ^{15}N -labeled species overcame the complication of fast nitrogen nuclear relaxation. Yin used a rate-equation analysis to interpret the results. Published data are of the highest quality. Yin extended the rate-equation method in paper [36].

In the decade after the National Biomedical EPR Center was founded, bridges with a continuous array of microwave frequencies from 0.5–8 GHz with discrete frequencies at 9.5, 18, and 35 GHz were built or purchased; a world-leading capability, saturation recovery, had been built; a new class of resonators—the loop gaps—were introduced; and about 50 papers per year were being written. I felt I was meeting my obligations to the National Institutes of Health to advance the field of EPR.

5.3. Multi-Quantum EPR

By mid-career, I had been wrestling for many years with the problems and benefits created by high-frequency (100 kHz) magnetic field modulation. I decided to make a serious effort to find a way to produce pure absorption or dispersion spectra without the use of field modulation as is routinely done in MRI. This decision led to a body of work that is grouped in Topic XII, Multi-quantum EPR. A total of 15 papers and reviews were written mostly between 1989 and 1995. A new category of EPR spectroscopy was created with good sensitivity and general purpose applicability.

The experiment involves irradiating the sample with two identical pure-tone time-locked microwave frequencies that are separated by, for example, 10 kHz. The spin system generates pairs of intermodulation sidebands that are symmetrical about the center frequency of the irradiating pair and can be characterized by emission and absorption of microwave quanta. Spectra can be formed from pairs of symmetrical sidebands by sweeping the applied magnetic field. For 10 kHz separation, sidebands at ± 15 kHz are labeled 3 quantum. At ± 25 kHz, the label is 5 quantum.

Paper [174] is a review that places this experiment in a historical context and builds the argument not only that it is new, but also that it has experimental parameters that are well suited to EPR spectroscopy. Paper [180] by Hassane S. Mchaourab and me describes a solution to Bloch equations using Floquet analysis that predicts under the assumption of sufficiently low incident microwave power that the 3 quantum sidebands will be proportional to $T_1 T_2^2$, while the 5 quantum sideband pair will vary as $T_1^2 T_2^3$. Paper [65] is an exhaustive analysis of the possibilities for active microwave devices to generate spurious instrumental sidebands that could confound the experiment.

Paper [181] applied multi-quantum (MQ) EPR to the mixed-valence copper site in nitrous oxide reductase at X-band, and 10 years later, Klug et al. applied it to the spin labeled spectrum of arrestin K257C at 35 GHz [183]. Excellent sensitivity was observed in both cases. Results were enhanced in comparison with conventional EPR methods by the greater dependence on relaxation parameters, which increases the spectral sensitivity to subtle features.

Although there are just two papers on biological samples, these results were considered impressive. The technique of pseudo-modulation [14, 15] was used to produce first derivative-like spectra, which were compared with field modulation spectra, and the results were intriguing.

I made a management decision that may have been unfortunate. The Q-band instrument was shifted in purpose to serve as the microwave source for a new spectrometer at W-band, and our Q-band MQ capability was lost. Now, as this is being written, Q-band MQ capability has been restored, and W-band MQ capability will be available very soon. We will be able to return to MQ spectroscopy, and we will have some additional assets. The technique of moving difference (MDIFF) [17] will have advantages with respect to pseudo-modulation. Simultaneous detection of dispersion and absorption MQ spectra will be possible. More importantly, we have already developed techniques for extracting information concerning very slow anisotropic rotational diffusion and can look forward to enhancing these displays by MQ methods. W-band seems important in these spin-label experiments to understand the motion of membrane-bound proteins because of increased overall spectral width. This multi-frequency MQ capability will be truly unique.

6. MRI RESEARCH

6.1. Diagnostic Radiological Human Imaging with Local Coils

My career was forever changed one day in 1984 when I had lunch with Thomas M. Grist, a medical student who was also trained as an electrical engineer. I found myself talking about LGRs, and he asked if they could be used in MRI. A quick calculation indicated that the ratio of an EPR sample size, say 1 cm at X-band, to the typical sample size of a person, say 1 m, was 100, while the ratio of radio frequencies was 10 GHz for EPR at X-band divided by 60 MHz at 1.5 Tesla (T), a ratio of 166—close enough—so I replied on the basis of this scaling argument, “yes.” We received one of the first commercial MRI scanners from GE in early December 1984. Patients were not planned until one month later, so we had a month to test Grist’s and my hypothesis. It worked truly well, and a “works in progress” paper was submitted to *Radiology* in June 1985 [304]. A virtual explosion of publications followed as a small army of radiologists found their way to our door. My name was on 36 surface-coil publications in the three years from 1986–1988 [Topic XXI]. We were asked to write five reviews, papers [304–308].

The principle that a surface or local coil should couple only to the tissue of interest for best image quality was established. Every anatomic region needed its own coil. It was obvious that we should start a company, and we did: Medical Advances, which was sold later to Intermagnetics General, which in turn was sold to Philips Medical Systems.

The radiologists had to relearn their profession. They were trained in the anatomy of dissection using visible light for contrast. MRI produces geometric slices and uses the variability of mobility of water molecules in various tissues to provide contrast. The long paper by W. D. Middleton et al. [349] carefully compares pathology in images obtained both by photography and MRI.

My coil team came to consist of J. Bruce Kneeland, MD, a radiologist with an undergraduate degree in physics from Stanford University, and Andrzej Jesmanowicz, PhD. Jesmanowicz was a physicist trained in NMR and also a refugee from Poland in the days of Solidarity. He somehow found his way to Milwaukee and became one of my closest colleagues. He was later elected to fellowship in the International Society of Magnetic Resonance in Medicine.

The team also had a trained research MR technologist and a radiofrequency (RF) technician who made coils. We had four hours of scan time each week on Monday nights.

There were some personal highlights that stand out in my memory from those days. One of these was the imaging of small parts, which included a special coil for imaging the eyelid [359, 361], another for fingers [350], another for carpal tunnel [340], another for the cornea and anterior segment of the eye [359], and another for skin [315]. The skin presented an interesting problem: large in two dimensions with a need to suppress depth sensitivity in the third. I was usually involved in the design of small parts coils.

Bloch had taught that there are two modes for NMR detection, U and V. They were orthogonal vectors at every sample point in the crossed coil configuration. I made a bimodal surface coil where the two crossed coils had essentially zero mutual inductance by symmetry, but the vector reception fields in general were not orthogonal [324] at a particular point in space. The physicists had fun with that. I am pretty sure that Bloch's condition that the dot product be zero everywhere is more restrictive than necessary—only the integral of the dot product is required to be zero.

Another interesting physics problem was brought to me by Scott J. Erickson, MD [354]. He had found bright spots near tendons in some images that did not correlate with pathology. It turns out that the description of the T_2 value of a tendon involves a dipole-dipole interaction that goes to zero at the magic angle. This is another example of the opportunities that can come to a physicist who is employed by a medical school rather than a research university.

We played a part in the widespread initiative to introduce multiple signal acquisition paths into MRI. Our papers are grouped in Topic XXIII. We did make the first publication in the field [323]. Although we did not actually have two channels available, we simulated two-channel reception by switching the signal path from one coil to the other. We entered the fray again in papers [330, 331], which allow simultaneous parallel acquisitions of different slices of data.

Coil patents include [422–425]. The drawings can be instructive for those who want to build their own coils. Functional MRI (fMRI) was discovered in 1991 and overwhelmed our surface coil program, as discussed in the next section.

6.2. fMRI and Resting-State Functional Connectivity MRI

My first NIH grant in MRI, which was primarily on surface coil development, included a specific aim on the purchase of a small local gradient coil for imaging of the hand and wrist from GE. GE eventually declined to deliver, and I asked my new graduate student Eric Wong to design and construct such a coil. He did this with spectacular success, producing some of the best images ever seen of the complicated structures of the hand [321]. There are potentially some serious safety issues in the placement of a local gradient coil in a large static magnet field. Torques on the support structure of the coil must cancel, and modes of failure must be carefully analyzed. This had been GE's concern, and we were very careful.

Wong was an MD/PhD student. He had already completed his first two years of medical school when he came to me. It seemed natural to consider a dissertation topic in

neuroscience. I suggested that he consider making a local head gradient coil with a local head RF coil with a view toward improved imaging of diffusion of water molecules. He designed and made both the gradient coil and a novel head RF coil, which employed a mirror (i.e., copper plane) at the top of the head. The coil extended inferiorly to about the level of the eyes. It was a so-called “birdcage” design. He also wrote gradient recalled echo planar code, which was known but perhaps not available anywhere else in North America. These were heroic achievements in the advance of imaging technology. See patents [426, 427].

At an ISMRM meeting, we heard rumors of success in blood oxygen level dependent (BOLD) contrast in human imaging. By “we” I mean Wong, his fellow student Peter Bandettini, and me. We realized by chance that we had everything in place to do the experiment. Success was immediate. Our paper was the first to appear in the field of fMRI [370]. A second paper on signal processing methods followed [371]. We have never claimed that we were first to have the idea, but apparently we were first to have publishable data.

The basic algorithm was to move the fingers of one hand in a self-paced manner for 20 seconds followed by 20 seconds of rest, repeating three or four cycles. A response is seen in the opposite side of the brain in the region of hand representation when the time course of images that include this region is analyzed. Each voxel time course is cross-correlated with the task boxcar waveform of on/off cycles. (It reminds me of field modulation in EPR [83].) An image set is then made by moving the fingers of the opposite hand, followed by the fingers of both hands. There is a lot of control in the experiment.

I recruited neuroscientists with expertise in various brain regions from across many departments of MCW, built a neuroimaging team, and published, published, published. I sensed a great opportunity [372–385]. We contemplated and then submitted a Program Project Proposal to the NIH, which was successful on the second try. In that submission, I became project director. Apparently, the NIH felt it was more important to have a technologist like me as head of the project than a neuroscientist at this particular juncture.

It was a golden era, with the best equipment in the world studying the most important object, our brains. The weekly works-in-progress meetings were amazing. Each year, we ran a one-week school. New and very strong students came our way including Allen Song, professor in Radiology, Duke University, and director of the Center for Brain Imaging and Analysis, and Bharat Biswal, about whom I will have more to say.

I recruited a mathematician, Robert W. Cox, who developed the software program AFNI (Analysis of Functional NeuroImages) first at MCW [296] and later at NIH in Bethesda, Maryland. Cox, Jesmanowicz, and I did a novel experiment—real-time fMRI [291]. Images were formed and signal intensity built up on the fly as data were acquired [291]. Bandettini was everyone’s colleague in laying out the foundations of the field. He also accepted a position in Bethesda. My students and colleagues were having an impact at the highest level of ongoing neuroscience research in America.

There was too much noise in the data. It was much higher in live subjects than in static phantoms. Biswal had come to me in a very insistent manner—he wouldn’t let me turn down

his application for admission to our program, even though his background was as an engineer and signal processor of images from satellites. I suggested as a dissertation topic that he acquire a data set from a subject at rest to see if he could make some sense out of the noise. He discovered that cross-correlation coefficients of pairs of time courses from opposite sides of the brain were very high. There is a synchrony of transient blood flow time courses that is revealed in a rich pattern that reflects the neural activity of the brain. Apparently, the basic BOLD mechanism that involves response of blood flow to an external task also is active in the brain at nominal rest because the brain never rests. A noted scientist in the field told me that this was the only truly new physiology that had been discovered by MRI methods. Our paper has been cited more than 5,000 times [399].

A biennial series of meetings on brain connectivity was established, and attendance has been approximately 500. A new research journal was established, *Brain Connectivity*. The Human Connectome Project was funded for about \$30,000,000 in 2009, followed by large specific grants for specific brain diseases. Although MCW was unsuccessful in competing for the large initial grant, we won two of the disease-specific grants, one on epilepsy (2015) and the other on Alzheimer's disease (2016). For a physicist like me, this is a dream come-true.

7. THE 21ST CENTURY

I became increasingly unhappy with the administrative burden of directing the Biophysics Research Institute and stepped down in 1998 in order to make additional time available for research. In the same year, I wrote review articles on saturation recovery [143] and ELDOR spectroscopy [127], which brought my experience in those fields up-to-date. In 2002, a two-volume Festschrift on my EPR work was written [69]. Although actually published in 2005, it provided a vast amount of information on my research interests and placed my work in a proper context. My goal in this final section of this brief autobiography is to provide an overview of my research since the new millennium began.

I was awarded a new grant in coil development, recruited Mett and Jason W. Sidabras (who had recently obtained an undergraduate degree in engineering with specialization in microwave engineering), and started a new initiative in resonator design with a focus on handling aqueous samples. I was able to obtain a renewal of the Center grant to extend our multi-frequency capability from 35 GHz to 94 GHz, or W-band, which enabled us to proceed with a totally new spectrometer development with considerable emphasis on advances in digital technology. On the MRI side, I decided to step back from human imaging and to develop imaging technology of the rat brain, where I perceived that there was a greater opportunity for advances in neuroscience. Finally, as things worked out, I was able to recruit a new EPR PhD graduate student, Kittell. Working together, we were able to find a solution to a very old problem in EPR signal acquisition: how high to detect pure absorption lines reliability, as is done in NMR spectroscopy, rather than the first harmonic response to applied magnetic field modulation. This was accomplished, and we called the new methods NARS spectroscopy, for non-adiabatic rapid sweep. It supplements our work on MQ EPR.

Access to our publications in these four open-ended initiatives is provided in the subsequent subsections.

7.1. Advanced Resonator Development

We introduced the concept of uniform field cavity resonators [44, 46, 47]. A sample tube that is placed along the axis of cavity mode is subject to co-sinusoidal variation of the microwave field. If the sample is responding linearly, there is no problem, but if saturation phenomena are present, the nature of the response changes along the axis, and the integral of the changing response blurs the detection of the nonlinear response. We proposed to solve this problem by creating a central section of the cavity that was at cut-off and strictly uniform in the so-called propagation direction. At the ends, other structures satisfied TE boundary sections, but the sample can be shielded from the changing microwave fields in those end structures. The concept was extended to LGRs [51] and in an article published in the current special issue of this journal the concept is extended to dielectric tube resonators (DTR). We seek to improve the quantification of nonlinear excitation in EPR spectroscopy.

In an MRI surface coil project for use in rat brain at 9.4T, the NMR frequency is 400 MHz, approaching the lowest frequency of the EPR LGR of 500 MHz. Two surface coil papers have been published recently in *Review of Scientific Instruments* by the Mett, Sidabras, Hyde group [318, 319]. The coil that is used is called “meta-metallic”—three turns of copper foil that is less than a skin-depth thick, separated by engineered layers of dielectric. This coil is self resonant. It is strongly coupled by mutual inductance to a second resonant surface coil, resulting in two modes. The mode where currents flow in the same circular direction is selected. The second coil feeds a capacitive network to a nearby low-noise amplifier. The quadratic equivalent circuit has been solved. It is claimed, with experimental results, that there is a factor of 8 improvement over a conventional one-turn surface coil. That would lead to a reduction in voxel volume to 75 microns cubic relative to our current standard of practice using echo planar imaging voxels of 150 microns cubic—a factor of two in each dimension. Noting that the cortical gray matter of a rat is about 1.4 mm thick, 20 voxels could be placed across the tissue, resulting in an excellent layer-specific investigation of vascular-neuronal coupling. A preliminary study at 200 micron resolution using a conventional surface coil has been published [407].

7.2. W-band

The new spectrometer incorporates an LGR of about a 100 loaded Q-value, or 1 GHz in 3 dB bandwidth. It is this remarkable bandwidth that makes the instrument novel. Frequency sweeps and frequency modulation papers have been published [71, 73]. It is a multi-arm bridge. The frequencies can be anywhere within the bandwidth. The instrument has saturation recovery capability [162]. It is believed to be the only high-frequency instrument in the world that can be used for saturation recovery. Saturation recovery was an enabling experiment for a sample involving a preparation from scarce human eye pairs from the eye bank [159]. W-band was important not because of the frequency but because of the exceptionally small sample volume—about 20 nL. A new type of automatic frequency control (AFC) is described in a recent paper [215], which allows long-term signal acquisition even though the Q-value is relatively low. Great attention was paid to path-length equalization of the arms as well as to minimization of leakage across arms. Fourier signal processing of the FID by rapid frequency sweep seems within reach.

7.3. 9.4T Small Animal MRI

We performed perhaps the first resting-state fMRI experiment in rat brain [405] and were among the earliest in task-induced fMRI experiments [348]. In a major initiative in collaboration with the Department of Plastic and Reconstructive Surgery, seven plastic surgery residents spent successive years in our imaging center working on cerebral response to forearm nerve injury and repair. Funding has been obtained and research is continuing. As is apparent in the topical bibliography, publications have been extensive, but some goals remain elusive. Pathways in the cerebellum, medulla, and thalamus that lie between the spinal column and the cerebrum have been difficult to identify. We are hopeful that this barrier will be overcome in the near future using the novel meta-metallic surface coil assembly described above.

7.4. NARS EPR Detection

The idea in NARS detection is to employ trapezoidal field sweep rather than sinusoidal field sweep at a frequency that is sufficiently high to avoid $1/f$ noise—say 20 kHz—with linear sweep rates and incident powers sufficiently low that FID does not occur. It is meant to be a nonsaturating low-power CW analogue. Fast digitization acquires repeated high-quality representations of a segment of the EPR spectrum. High-speed digitization and accumulation coupled with reasonable assumptions concerning the smoothing of data across regions of adjacent spectral points results in the final spectrum. The entire spectrum is formed by acquisition of segments, which might be only 1 spectral point apart. Actually, it is very much like sinusoidal field modulation, but with bandwidth narrowing occurring in the computer rather than the time constant of a lock-in detector. Without doubt, the lock-in detector is destined to drop from usage, since its function can be so easily replicated in software. The more advanced methods of NARS follow, and extension to acquisition of transient FID signals is natural.

A recent paper [76] applied NARS spectroscopy to a spin-label sample undergoing very slow rotational diffusion. Turning points revealed the onset of FID response, while regions between turning points responded normally. Thus, we would seem to have a way to obtain spectral information that is somewhat like that obtained by the transfer of saturation without the broadening that occurs in that experiment from magnetic field modulation.

Paper [17] applies NARS to a powder spectrum of Cu(II). The spectral features change quite a lot across the spectrum. Pseudo-modulation [14, 15] is a post-processing algorithm that mimics the effect of sinusoidal magnetic field modulation when it is applied to a NARS spectrum. The amplitude of the modulation is an input to the algorithm. It can be set to enhance inflections in the NARS spectrum for a closer look at a spectral region of particular interest by producing a derivative-like display. An alternative to pseudo-modulation, the moving difference or MDIFF algorithm, was introduced in the appendix of the paper [17]. MDIFF subtracts signal intensity of two points at a preset separation as it is swept across the spectrum and displays the results to arrive at a derivative-like display. Pseudo-modulation and MDIFF algorithms transform the spectra and also filter spectral noise, although in different ways. Proper use of them depends on *a priori* knowledge and conscious awareness

of the spectral information that is of interest. NARS acquisition provides a spectrum without major initial assumptions and allows refinement of post-processing analysis.

8. AWARDS

I was elected to fellowship in the American Physical Society in 1975 and received the degree of *Doctor Honoris Causa* from the Jagiellonian University in Krakow, Poland in 1989. I hold The James S. Hyde Professor of Biophysics chair at the Medical College of Wisconsin.

Major professional awards include the Bruker Prize from the Royal Society of Chemistry in 1989, the Gold Medal from the International EPR (ESR) Society in 1993, the Zavoisky Award from the Russian Academy of Sciences (Kazan) in 1995, and the Gold Medal from the International Society for Magnetic Resonance in Medicine (ISMRM) in 1999.

I became a fellow of the International Society of Magnetic Resonance (ISMAR) in 2008. The “James S. Hyde Lectureship” is awarded biennially by the Conference on Resting State and Brain Connectivity.

9. BIBLIOGRAPHY: A LIST OF EPR AND MRI PUBLICATIONS AND PATENTS BY TOPIC

Following is a comprehensive list of papers, book chapters, reviews, and patents either authored or co-authored by me during the period of 1960 to 2017. It is organized in two sections, EPR and MRI (including MRS). There does exist a certain linkage between EPR and MRI sections concerning resonators, and readers interested in this subject may wish to look over the titles in both sections. In addition, papers on vision research, both EPR and MRI, are grouped in the MRI section. Whenever feasible, each topic is introduced by reviews. The organization of topics tends to be chronological, with steadily increasing use of digital technology and computers. The topics are identified by roman numerals. Within topics, the publications are listed in chronological order. A brief summary is provided for each patent listed. With only a few exceptions, all of my publications and patents are listed, and some are listed more than once when there are two or more appropriate topics—for example, a technical contribution and an application. Several publications that bare my name but do not fit the topics are omitted. Several other publications are omitted if access seems limited. Publications are numbered continuously, and publications that appear more than once carry the first number used.

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