Report on the 2015 NHMFL User Advisory Committee meeting
Held in Santa Fe/Los Alamos, NM, from October 27th to 29th, 2015

Chair: Chris Wiebe, Department of Chemistry, University of Winnipeg/University of Manitoba (adjunct, Department of Physics and Astronomy, McMaster University)
DC/Pulsed/High B/T Vice-Chair: Madalina Furis, Department of Physics, University of Vermont
NMR/MRI/ICR/EMR Vice-Chair: Robert Schurko, Department of Chemistry and Biochemistry, University of Windsor

User committee members:

DC/PFF/High B/T committee: Chuck Agosta (Clark University), Kirsten Alberi (National Renewal Energy Lab), James Analytis (University of California, Berkeley), Kenneth Burch (Boston College), Jason Cooley (Los Alamos National Laboratory), Nathanael Fortune (Smith College, Executive Committee Member), Madalina Furis (Chair for DC/ Pulsed Field /High B/T, University of Vermont), Malte Grosche (Cambridge University), Zhigang Jiang (Georgia Institute of Technology), Lu Li (University of Michigan), Philip Moll (Max Planck Institute for Chemical Physics of Solids), Jeanie Lau (University of California, Riverside), Jamie Manson (Eastern Washington University), Wei Pan (Sandia National Laboratories), Filip Ronning (Los Alamos), Chris Wiebe (University of Winnipeg, User Committee Chair), James Williams (University of Maryland)

NMR/MRI committee: R.W. Schurko (Chair, University of Windsor), Marek Pruski (Ames Lab, Iowa), Michael Harrington (Huntington Medical Research Institute), Brian Hansen (University of Aarhus), Eduard Chekmenev (Vanderbilt University), Oc Hee Han (Korea Basic Science Institute), Doug Kojetin (Scripps Research Institute), Len Mueller (UC Riverside), Fang Tian (Penn State University), Tatyana Polenova (University of Delaware), Scott Prosser (University of Toronto)

EMR committee: Kurt Warncke (Emory University, U.S.; Chair), Chris Kay (University College, U.K.), Dane McCamey (University of New South Wales, Australia), Christos Lampropoulos (University of North Florida, U.S.), Stefan Stoll (University of Washington, U.S.)

ICR User Advisory Committee: Jonathan Amster (Franklin College), Michael Chalmers (Eli Lilly and Company DCR&T Analytical), Michael Freitas (Ohio University Medical Center), Elizabeth Kujawinski (Woods Hole Oceanographic Institution), John Shaw (University of Alberta), Forest White (MIT)

On behalf of the User Committee, we would like to express our deep sympathy to the family of Glenn Nix, who recently lost his life in a tragic accident just a week before the User Committee meeting. In these difficult times, we commend the NHMFL for taking active steps to confront the safety issues related to this event and to avoid its recurrence. We would furthermore like to emphasize that the NHMFL's safety record has been very good over the long term, and it is our view that the lab has the user's safety at the forefront of its mission.

We would like to thank the Los Alamos branch of the NHMFL for hosting a well-attended and informative User Committee meeting. All three branches of the NHMFL participated and played a role in organization. The general consensus of the user community was that the meeting was a great success, with highlights being the articulation of the future Science Drivers, the tours of the Los Alamos laboratory, and the careful consideration given to responding to issues that arose from our last report. We are very confident that all three branches of the magnet lab will continue to be involved in transformative research across many disciplines, and we are excited and optimistic to see how the three sites will evolve in the future.

(1) Executive summary

Before addressing the particular issues which arose from the various subcommittees, we would like to first discuss general developments which affect all of the subcommittees at the NHMFL (and the broad user community). The remainder of the report details specific issues which are unique to the different subcommittees.

(i) Recompetition/Renewal

The User Committee was very pleased to hear that the recompetition is now a renewal proposal. We see this as a very positive development. However, there is a considerable amount of work left to complete with the renewal project. The NHMFL has taken this task extremely seriously, and we were excited to hear about the new Science Drivers and the new projects proposed in the breakout sessions. As a community, we would like to refine some of these Science Drivers more, and also have time to consider the many directions that the NHMFL can take in the future. We are confident that together we can articulate the future vision of the magnet lab. We are pleased with the general directions of the Science Drivers as they were presented at our meeting.

(ii) Housing

The User Committee emphatically supports the new housing project at FSU for users. We would like to give the strongest possible endorsement for an immediate solution to the housing problem in Tallahassee for users. Many national laboratories have housing in place for external users that make travel for experiments more feasible. The NHMFL needs this, and we strongly recommend that the NSF provides support for the proposed housing project. The housing problem is less of an issue at the UF and Los Alamos sites (although we did hear from users that readily available parking passes from UF might be useful in the future).

(iii) Future hiring strategies

The recent hires at the NHMFL (and the proposed hires in the future) are to be commended. This was a chief concern of the UAC from our last report, and we are very encouraged to see the NHMFL following through with their commitments to having the best faculty and support available. In particular, the hiring of Dr. Laura Greene as Chief Scientist is an excellent addition to the condensed matter physics capabilities of the lab. We are confident that she will be able to provide the significant leadership and vision in the future. Director Boebinger also detailed other strategic hires across many disciplines that we find exciting and necessary for the renewal

proposal. The UAC was very pleased to hear about a long range plan for the hiring and retaining talent at the NHMFL.

(iv) Diversity

The User Committee was very impressed by the detailed report given by Director Boebinger for increasing diversity at the NHMFL. There are many promising future developments, including the new five part plan, and initiatives such as the American Physical Society's MS-to-PhD Bridge program. While we still understand that there is room for improvement in increasing diversity at the NHMFL, we acknowledge that the magnet lab is aware of many of the issues and is working to make increasing diversity one of the top priorities.

(v) The new website

The new website is visually appealing, informative, and overall very well done. This is reflected in many of the metrics that have been used to track how users are increasingly seeing the value of the new site and how information is processed. The work is truly excellent, and we see the website as a model for how national laboratories can help users and the general public learn more about magnetism and the work done at the magnet lab. We have had very good feedback from the user base, as well, concerning the changes, and in particular how many experiments can now be planned with ease from the new drop down menus.

(vi) Safety

On behalf of the UAC, we would like to again express our condolences with respect to the recent tragedy at the magnet lab in Tallahassee. The UAC applauds the response of the NHMFL as compassionate and pro-active. An independent Investigating Committee has been convened to determine the cause of the accident and make recommendations for changes in the future. We believe the approach of the NHMFL to suspend operations on both in-house magnet development and DC program operations until the end of the scheduled shutdown to be appropriate. The UAC found the update on safety initiatives, protocols, and success stories to be exceptional. While there is always room for improvement in terms of safety at national facilities, we feel that the NHMFL is leading the way for safety standards.

(vii) Summer school and outreach

The UAC is continually impressed by the tremendous educational activities which are organized every year by the NHMFL. The summer schools are essential as training programs for future users and have a great impact in the user community. We are also very happy with the excellent outreach activities from all three branches of the magnet lab. Keep up the spectacular work!

(viii) User committee changes

The UAC discussed minor changes to the bylaws that were completed over the last year. There was also a discussion of the protocol to maintain confidentiality among scientists conducting experiments at the NHMFL. In general, users at the NHMFL feel that their data is secure.

(2) Report on the DC/ Pulsed Field /High B/T Facility

Contributors to the DC/ Pulsed Field /High B/T report:

The committee is comprised of:

Chuck Agosta (Clark University)
Kirsten Alberi (National Renewal Energy Lab)
James Analytis (University of California, Berkeley)
Kenneth Burch (Boston College)
Jason Cooley (Los Alamos National Laboratory)
Nathanael Fortune (Smith College, Executive Committee Member)
Madalina Furis (Chair for DC/ Pulsed Field /High B/T, University of Vermont)
Malte Grosche (Cambridge University)
Zhigang Jiang (Georgia Institute of Technology)
Lu Li (University of Michigan)
Philip Moll (Max Planck Institute for Chemical Physics of Solids)
Jeanie Lau (University of California, Riverside)
Jamie Manson (Eastern Washington University)
Wei Pan (Sandia National Laboratories)
Filip Ronning (Los Alamos)
Chris Wiebe (University of Winnipeg, User Committee Chair)
James Williams (University of Maryland)

(i) General comments:

The user committee commends the DC/Pulsed Field/ High B/T facilities for the superb effort made to respond to all committee recommendations from the previous year. We would also like to once again state that the support for the user community was excellent over the last year, and we are continually impressed by the level of professionalism displayed by all three branches of the magnet lab. In particular, we were very excited by the progress made at the DC facility with the 32 T high T_C superconducting magnet, the progress on a 40T / 28MW resistive magnet development and the projected opening of the third bay in Gainesville to users. The UAC also applauds the DC facility's focus on allowing new capabilities that extend the available experiments at high field to include spectroscopic probes including NMR (relevant to understanding quantum matter and molecular biology), IR/Raman (probing dynamics of quantum and biological systems with symmetry specificity) and scanning tunneling probes (allowing, for the first time, atomic resolution of the magnetic response of a myriad of materials when the magnetic field is on a comparable energy scale to other interactions in the system.)

We would like to reiterate that one of the chief concerns that the DC field facility has is with finding more time in fields up to 40 T and beyond. The 45 T hybrid, for example, is still very much oversubscribed. The 40 T resistive magnet should be able to handle some of these experiments, but long term planning of the magnet lab should include alternatives to reaching these fields (in case either of these systems break down, for example). We feel that this should be a key part of the renewal proposal.

The progress made in the development of a pulsed magnet system with a space-free –optics compatible tail that would operate in a horizontal geometry is fantastic. The committee believes that this will greatly contribute to possibly creating a "spectroscopy cluster" at the lab. This cluster would encompass complementary spectroscopy techniques such as NMR, EPR, ESR, MCD, cyclotron resonance and other flavors of optical, far infrared and THz spin-resolved ultrafast measurements that altogether address electronic systems with complex thermodynamic behavior. This magnet will also be great alternative to users that seek optical spectroscopy measurements compatible with fast acquisition times, significantly relieving some of the pressure currently existing on the DC HELIX magnet scheduling.

The committee was very excited to learn about the plans to build dedicated user housing units next to the Tallahassee laboratory and expresses its gratitude towards the Florida State University leadership who made this possible. The housing situation is truly very critical and the overall modest increases in NSF single investigator grant budgets compared to the rising cost of travel and housing on the open commercial market are really felt as an increasing burden by the users. The committee also stresses the need for a temporary solution until the new building becomes functional. The alternative would be a substantial increase in traveling budgets for single investigators that are DC facility users.

On behalf of the NHMFL users, the committee members extend their condolences for the most unfortunate work-related accident that resulted in the tragic loss of life for a member of the DC facility team. This came as a great shock to all of us especially since, as users, we always experience the lab as a very safe working environment with excellent risk management and safety procedures in place. The users are regarding this as a most unfortunate incident and encourage the lab to continue their excellent work in identifying potential risks and periodically updating their policies and procedures on safety. We would like to reiterate as well that the community understands that the incident happened during a construction operation at the NHMFL, and not in an area where users would conduct experiments.

The committee endorses the PFF's innovations in both extending its range of available magnetic fields to 150T and in employing nanofabrication techniques to allow more experiments. From the perspective of the UAC it is important to stay competitive internationally both in making higher magnetic fields available and in making new experiments possible. The utilization of Focused Ion Beam micro-structuring and other nano-fabrication facilities will enable users to measure quantum transport properties on materials with extremely low resistivity (ultra-clean materials). In addition, it may enable faster rise-time magnets to be useful by significantly reducing sample heating during a pulse, by reducing the volume of the material probed. Experiments designed for a faster rise time magnet could facilitate the use of smaller magnets, which are easier to replace and can pulse significantly more frequently. This in turn will allow a much greater throughput of experiments and a faster (and cheaper) turnaround of magnets as they approach the end of their lifetime.

In last year's report, we commented on the need for enhanced communication between the PFF and DC/High B/T committees. Many of the materials and problems addressed by both communities have a fair deal of overlap. Even though the PFF and DC/High B/T subcommittees

had separate break out sessions at the UAC meeting, we felt that there was good communication between both groups. There is always room for improvement, though, and we feel that we can enhance communication and collaborations in the future.

The committee also endorses future Science Drivers to enhance the current limits of pulsed and DC fields at the magnet lab, to explore new techniques and improving experimental capabilities, and to increase field homogeneity. These will open new fields of research and enable truly cutting-edge science to be completed at the magnet lab. We look forward to playing a role in shaping the Science Drivers in the Renewal proposal.

(ii) Priority list of recommendations and executive summary:

- We encourage the development of new capabilities at the NHMFL, including the 32 T high T_C superconducting magnet, the 40T/28MW resistive magnets, and the 3rd experimental bay in Gainesville.
- The UAC is very pleased to hear about the new housing units in Tallahassee for users, and the support given by Florida State University towards this goal. We recommend that the NSF also supports this new initiative.
- The optics program is developing well and the possibility of a "spectroscopy cluster" is exciting and novel. Additional support may be required through new positions.
- We are still developing a way for the PFF and DC communities to interact in a more constructive manner to encourage more synergy between the subcommittees. This will take time and communication. The two subfields are highly related to each other and have worked well together in the past.
- We continue to recommend future developments of higher fields (up to 150 T) and new techniques at the PFF and nanofabrication facilities.
- The User Committee is encouraged by the new Science Drivers mentioned at the meeting. We support the general themes expressed in the Renewal proposal, and we would like to play a part in shaping these themes in the future.

Report of the Magnetic Resonance Division User Committees
Sections: I. NMR and MRI, II. EPR and III. ICR

I. NMR and MRI

NMR/MRI UAC and contributors to this section of the report:

At UCM:
Robert Schurko (Chair, Windsor)
Eduard Chekmenev (Vanderbilt)
Michael Harrington (Huntington Medical Research Institute)
Brian Hansen (Aarhus)
Oc Hee Han (Korea Basic Science Institute)
Fang Tian (Penn State)

Remote participants:
Linda Columbus (Virginia)
Doug Kojetin (Scripps, FL)
Len Mueller (UC Riverside)

1. Overview. The Users' Advisory Committee for the MR division (hereafter referred to as the UAC) is impressed with progress made at the MagLab in terms of magnet technologies, probe construction, publication quality and count, and recruitment and outreach activities. High-field NMR and MRI facilities are providing research groups from the United States and around the world with access to unprecedented experimental capabilities, and have supported major research initiatives in materials science, structural biology and MR imaging. The UAC *strongly endorses* investment in new personnel (especially in probe development and maintenance), and support for acquisitions and developments of consoles and magnets. The MagLab is still the world leader in high-field MR methods – an enormous investment has already been made in this successful institute, and continued investment must be made to maintain this position of leadership.

2. Personnel. The UAC is concerned about hiring of technical staff in the MR division. Last year, it was recommended that an rf engineer be hired for probe development. The current workload of building and designing new probes (e.g., for the new SCH system), as well as maintaining all of the active probes for all of the current systems (i.e., DNP, MRI), lies with Peter Gor'kov (team leader), Jason Kitchener (probe engineer) and Richard Desilets (machinist). There are only two indirectly involved support staff, Ilya Litvak (Research Assistant, SCH project) and Steve Ranner (Research Engineer). The plans for probe development for all of the NMR and MRI systems (including new systems under development) are very impressive (*vide infra*); however, additional support is desperately needed in this area - the workload is much more than the current team can manage. The UAC recommends the hiring of two rf engineers for probe maintenance and development: one for solid-state and solution NMR and one for MRI.

The UAC is also concerned that a replacement for Rafael Bruschweiler was not found over the past year, as this hire is critical for both FSU and the MagLab. However, we are encouraged to hear that there is a future faculty line for this position, and the development of an associated 30 T NMR spectrometer is in progress for future research on intrinsically disordered

proteins, metabolomics, and protein dynamics. If this new faculty member is hired in the next year, it is crucial that an 800 MHz NMR spectrometer be offered as part of a start-up package, since there will be some wait for the completion of the aforementioned 30 T system.

The UAC believes that a replacement for Anant Paravastu should be found (he departed for Georgia Tech this past year). A faculty member with similar research interests (biomaterials, biopolymers, amyloid fibers, etc.) would be an active external user of the MagLab facilities.

Finally, the UAC is encouraged by the recruitment of Matt Merritt to UF, who will aid in growing research efforts in DNP and in vivo NMR spectroscopy. In addition, while it is unfortunate that Art Edison departed for the University of Georgia, it is good that a strong working relationship will exist with the MagLab for continued work on HTS probes.

3. Equipment, Infrastructure and Use of Facilities.

Intro. The total count for active NMR and MRI instruments associated with the MagLab is 21 (13 at FSU/MagLab, Tallahassee and 8 at UF/AMRIS, Gainesville). Magnetic field strengths vary from 3.0 to 21.1 T, with an incredibly wide range of capabilities for solid-state NMR, solution NMR and MRI. Research areas and applications include SSNMR of materials and biosolids, biological solution NMR (e.g., proteins, metabolomics, clinical, in vivo) and (micro-)imaging of samples, animals and humans. The installation dates for these spectrometers range from 1994-2013, and numerous recent upgrades have been made at both locations. Exciting new technologies are being developed, including HTS and SCH magnets for ultra-high field NMR spectroscopy, new DNP NMR spectrometers (both for dissolution in solutions and solid samples), and numerous specialty probes for NMR and MRI (vide infra). Given this impressive array of equipment, and many of the associated technologies that are unique to the MagLab, the UC makes one of its strongest recommendations: funding and resources must be put in place to not only maintain the aforementioned systems, but also to support the acquisition of new systems and the development of new hardware (i.e., magnets, probes and gradients).

Highlights - FSU. Several research highlights across areas of solution and solids NMR, as well as MRI, are mentioned, including the determination of protein structure in the *M. tuberculosis* cell division apparatus (Cross group, FSU), in vivo ^{35}Cl and ^{23}Na MRI of rat glioma (Schepkin *et al.*, NHMFL), the use of ^{35}Cl SSNMR to probe polymorphism in active pharmaceutical ingredients (Schurko group, Windsor, Canada), and ^{17}O SSNMR of low-barrier hydrogen bonds in nicotinic acid (G. Wu, Queens, Canada). The UAC was also very impressed with the development of the first facility in the U.S. to perform SSNMR experiments at temperatures up to 750 ^{o}C (Y.Y. Hu, FSU), and continued probe development for the new 36 T SCH instrument, as well as other spectrometers (*vide infra*). The UAC feels that support is needed for: (*i*) Hiring of additional personnel for probe construction and maintenance (see *Sec. 2*). (*ii*) Equipment and Supply/OPS budgets (these budget lines have been flat for years, and increases are desperately needed). The UAC is surprised that the NMR/MRI groups have little access to overhead funds, and must continually make requests for even small amounts of funding (ranging from $1-5 K). Furthermore, in reviewing MagLab reports from 2013 and 2014, it seems that the MR division only receives ca. 15-20% of the total budget, while accounting for ca. 30% of output in terms of publication count. (*iii*) A new console for the flagship 21.1 T NMR system, including new gradients, amps and shims (for enhanced resolution for NMR/MRI experiments).

Highlights - AMRIS. Two major research highlights are mentioned, including work by the Mareci lab on new contrast types generated by combination of high resolution diffusion

weighted imaging and modelling (featured on the cover of Magnetic Resonance in Medicine), and MR microscopy work by the Blackband group on fly brain allowing anatomical segmentation of the drosophila brain in 3D (which received media attention). The UAC also highlights the following issues: (*i*) The 750 MHz WB is oversubscribed by users, indicating that this instrument is of great importance to many research groups. (*ii*) Staging equipment for the 600 MHz magnet for *in vivo* imaging would significantly reduce the load on the 750; this is a good plan for expansion and maximizing the return on investment. (*iii*) Microimaging of cells and tissues is leading the field; however, Bruker coils on high field magnets are often not working. In-house design and construction of rf-coils and gradients may be the best option.

Publications, Usage & Funding. The FSU team reports 61 publications in 41 different journals (including 2 in JACS, 2 in PNAS and 1 in Nature Chemistry), and 71 different credited grants (25 from NSF, 21 from NIH and 25 other sources, including internationals). Most of the publications are associated with the 21.1 T instrument. In addition, they report the participation of 106 senior personnel, 34 PDFs, 80 students and 9 technicians. The UF/AMRIS team reports 45 publications, and 233 users, including 81 senior personnel, 30 PDFs, 85 students and 15 technicians. The FSU and UF/AMRIS teams report 3347 (for 2015) and 1653 (for 2014) user hours, respectively.

User Recruitment & Outreach. The UAC is pleased to hear that recruitment and outreach activities continue at both facilities. The new MagLab web site is extremely attractive and well-organized, and has been very popular for regular users and new recruits to the facility, as well as to the general public. At FSU, an open house day was well attended, numerous tours of the MagLab facilities were offered, and a biological SSNMR winter school was offered. The UAC is happy to hear of possible plans for future winter schools in SSNMR of materials and MRI methods and applications. At UF/AMRIS, workshops on SECIM metabolomics (60 participants) and real-time FI and neurofeedback (200 participants) were offered. AMRIS is planning a future coil building workshop as well. Twenty groups toured the AMRIS facilities, and activities are offered in undergrad and K-12 research, as well as graduate student (80) and PDF (35) training. The UAC recommends that recruitment and outreach activities are continued and strengthened, for the purposes of increasing the user base and related scientific output, and for attracting young people to research in chemistry, biology and physics.

4. Magnet technologies and probe development.

Magnet technologies. As discussed in previous reports, an important feature of the MagLab which places it ahead of all other facilities in the world is the continued development of new magnetic field technologies that will be transformative in the way that NMR spectroscopy is utilized. In particular, the Keck (25 T), Platypus (30 T), SCH (36 T) and NHMFL Hybrid (45 T) magnets and spectrometers all present different degrees of field strength and resolution that will enable a broad variety of applications. There was not a large focus on new developments in this area at this year's UCM (rather, focus was upon consoles and probes, *vide infra*), with the exception of the 36 T SCH, which is coming online in mid 2016. This system currently has a field homogeneity of 1 ppm (^{1}H, 1.59 GHz), and will be used for an enormous variety of experiments over the coming year. The UC is impressed by the potential for experiments on quadrupolar nuclides in inorganic and organic materials, investigations of biological samples, and unprecedented developments in MRI at this field. The UAC would also like to point out that imaging gradients will need to be developed for the SCH system, very likely in collaboration with external partners.

Probes. At this year's UCM, much discussion of new technologies for NMR and MRI focused upon probe development, an area lead by Peter Gor'kov (NHMFL, Tallahassee). One of the most important developments is tune cards, which can be inserted into probes to tune them over broad frequency ranges. This is crucial for probe use at high magnetic fields, due to the great variation of frequency ranges for nuclides across the periodic table. The UAC feels that this technology could be a real game-changer for probe development around the world, and perhaps inspire a new generation of broadband probes for both NMR and MRI purposes. The UAC is also glad to hear of the development of several probes for the 36 T SCH: (*i*) A 2.0 mm HXY indirect direction MAS probe has been developed with a sensor for magnetic field regulation, external lock signal and passive ferroshims – this is crucial for operation under conditions of fluctuating B_0 fields. (*ii*) A 3.2 mm single-channel low-gamma MAS probe has been built with operation between 50 and 280 MHz, which will be ideal for the study of many quadrupolar nuclides that cannot be studied at lower fields.

5. DNP. The NMR/MRI group at the MagLab has done an excellent job in getting the DNP program off the ground. This is a titanic effort given the engineering and the instrumental (cost and complexity) requirements. There are no focused programs on hyperpolarization (including DNP) at NSF, NIH or DOD to the best of our knowledge; however, there are targeted programs receiving tremendous support in Europe and the U.K. The UAC feels that the DNP program at NHMFL will face fierce global competition, and must act to remain ahead of the pack. Since hyperpolarization/DNP techniques require deep expertise in magnetic resonance and physics (two areas of enormous strength at the NHMFL), the MagLab should play a leading international role in this area.

The UAC is pleased with a number of developments, including: (*i*) the hiring of Matt Merritt (UF) to play a leading role in the DNP program. (*ii*) Clear plans for developing a number of rf probes for use in DNP/hyperpolarization experiments. (*iii*) Insuring that there is operational DNP equipment, and planning for the acquisition of more instrumentation in the future.

The UAC encourages significant expansion of the modes of hyperpolarization at both sites and increased synergies between the Tallahassee and UF groups. Specifics include: (*i*) MagLab teams should pursue all current and suggested plans for DNP program. (*ii*) Hiring of additional faculty and staff (engineer and technician) to support the expansion of hyperpolarized NMR/MRI program. (*iii*) Installation of parahydrogen generators (commercial or home-built) at UF and Tallahassee sites. (*iv*) Leveraging of existing faculties at UF (e.g., Russ Bowers) to develop other hyperpolarized modalities at the MagLab facilities. (*v*) Support of a collaborative program between hyperpolarized NMR/MRI users and the high B/T facility. (*vi*) Establish optical-pumping Xe hyperpolarization capabilities at UF and/or Tallahassee site. These activities require significant investments in personnel, equipment and maintenance; again, if the NHMFL is to maintain world leadership in polarization techniques, this support is well justified.

6. MRI. The UAC was pleased to learn of a variety of developments and advances in MRI research and equipment development (see also the AMRIS section above), including high resolution imaging of live (perfused) brain slices (Blackbland) which is expected to allow studies that can inform interpretation of clinical MRI. The UAC is impressed with plans to support 9 cm small-animal MRI at 30 T within the SCH magnet program (this is remarkable, since the nearest commercial small-animal MRI system is limited to ca, 15 T). In addition, the idea to have a stage 600/14T MHz MRI capability probe(s) for performing preliminary experiments before

working at the higher fields is an outstanding initiative. This will save a lot of experimental time at higher fields, increase the throughput for external users, and serve to attract new users. It is also highly synergistic with dissolution DNP program and hyperpolarization efforts in general.

The 900 is still the world leader for *in vivo* MRI. Upgrades to the original console can be expected to extend the sensitivity of the MRI experiment. In addition, plans for a wider bore (89 mm) SCH magnet for imaging are very exciting, as this will enable unprecedented sensitivity for functional sodium MRI. Increased spatial resolution in many MR modalities will enable new discoveries in brain imaging as it will extend focus to more prevalent but less structurally severe pathologies (concussion and dysfunctional brain disorders) in rodent models of disease.

The UAC again emphasizes that it would be a great advantage to hire additional support staff to support MRI probe development. The probe team mentioned in *Sec. 2*, along with Bill Brey, Sam Grant, Victor Schepkin and Jens Rosenberg, provide great user support for the MRI. An additional probe engineer to assist in the construction of multi- and single-nuclide probes (e.g., for rats/mice, and in vivo without anesthetic), is regarded as a top priority.

7. Other Items. The UAC is very happy to hear of the very positive responses from users who have interacted with the staff in the MR division of the Maglab. In addition, the UAC is pleased to hear that a new residence for MagLab visitors is underway in Tallahassee. This will make a major difference for visiting scientists, grad students and PDF, and certainly encourage increased usage of the MagLab facilities.

8. Priority List of Recommendations and Executive Summary

In summary, the UAC is impressed with progress over the past year in the MR division of the MagLab. In order for the MagLab remain the preeminent NMR, MRI and DNP NMR facility in the world, we believe the following recommendations must be considered.

- Continued support for acquisition and maintenance of spectrometers and consoles. In particular, support for a new console for the flagship 21.1 T spectrometer is a top priority. We emphasize that funding must be in place to support and expand the most impressive fleet of NMR spectrometers in the world.
- The hiring of two rf engineers for probe maintenance and development: one for solid-state and solution NMR and one for MRI.
- A replacement faculty position for Rafael Bruschweiler in structural biology. An 800 MHz NMR spectrometer will be needed for this faculty member during the wait for access to the new 30 T spectrometer.
- A replacement faculty position for Anant Paravastu in the areas of materials, biomaterials, biopolymers and/or amyloid fibers.
- Expanded support for development and acquisition of probes and gradients (both NMR and MRI).
- Increased *Equipment* and *Supply/OPS* budget lines.
- Access to overhead funds to reduce the administrative burden and costs associated with requesting small amounts of support funding (e.g., 1-5 K).
- Expansion of DNP/polarization research is crucial; in order to maintain an international leadership position in this area, investment must be made, as many impressive new laboratories and facilities are active around the world.
- The MagLab should continue to maintain a healthy working relationship with Bruker, for both acquisition of new equipment and future exchange of IP.

II EMR

EMR UAC:
Kurt Warncke (Emory University, U.S.; Chair)
Erik Cizmar (P. J. Safarik University, Slovakia)
Christopher Kay (University College London, U.K.)
Dane McCamey (University of New South Wales, Australia)
Christos Lampropoulos (University of North Florida, U.S.)
Stefan Stoll (University of Washington, U.S.)

Program, Director and Personnel

Program

- Overall the EMR UAC was very impressed with the accomplishments of the Electron Magnetic Resonance (EMR) program in the past year.
- The EMR program continues to be a world-leading high-field (HF) EMR facility.
- The productivity is outstanding. The EMR group had 38 publications in the preceding period (2014), and is on pace in the 2015 period to match or surpass this mark.
- The EMR user base is strong, with 143 to date. The significant increase seen in 2013 (144) was sustained.
- The user program is heavily utilized (oversubscribed).
- A user survey conducted by the UAC just prior to the meeting indicates great satisfaction with the current program.

Director

- The EMR director, Stephen Hill, shows highly effective leadership, combining in-depth technical expertise and knowledge of the multi-disciplinary science being performed.
- The EMR director displays a high degree of commitment and responsiveness to user needs, and to user-driven scientific directions.

Personnel

- EMR staff (Jurek Krzystek, Andrew Ozarowski, Likai Song, Johan van Tol) have excellent technical expertise and, by using their respective instruments, are driving the science in their areas.
- Postdoc Sebastian Stoian has made strong contributions to EMR and Mössbauer spectroscopies.
- Postdoc Thierry Dubroca (hired by Hill; originally supported by Hill and NHFML) makes innovative, critical contributions to the Dynamic Nuclear Polarization (DNP) effort.
- Engineer Bianca Trociewitz (hired by Hill; originally supported by Hill and NHFML) has made innovative, strong contributions to DNP and to the HF CW-EPR (17 T) spectrometer.
- University of Florida (UF) faculty Alexander Angerhofer and Gail Fanucci continue to make strong contributions to the program, as science drivers, and by interfacing with the other magnetic resonance programs
- New Hire, Postdoc Johannes McKay (from the group of Graham Smith at St. Andrews; builder of the HiPER instrument) is an exceptionally well-targeted hire. He brings expertise about HiPER to the NHMFL.

Instrumentation

HiPER W-band EMR spectrometer

- The high power (1 kW) amplifier and associated components (including laser with a silicon switch, as well as an Attocube drive) were installed in August, 2015. This was the top-priority recommendation by the UAC last year. This is an excellent completion of the HiPER capabilities, enabling pulsed experiments with significantly increased sensitivity.
- For HiPER, thin-layer sample holders for solution samples were developed, in response to the request of users. These holders out-perform commercial W-band sample holders by an order of magnitude in concentration sensitivity.
- The UAC is pleased that the first data obtained on the HiPER spectrometer, with co-author Fanucci, has been been submitted for publication.

HF (17 T) CW transmission EMR spectrometer:

- The aging 180 A power supply for the 17 T magnet was replaced. This was a crucial and top-priority recommendation by the UAC last year. The new power supply provides renewed, reliable operation.

HF pulsed quasioptical EMR spectrometer:

- Routine operation continues with high sensitivity up to frequencies of 240 GHz. Operation to 330 GHz is possible with reduced sensitivity (limited source power).

SCH, 35 T Magnet System:

- A transmission probe for the 35 T magnet was designed and constructed in a collaboration between NHMFL (staff members, Krzystek and Trociewitz) and the Dresden High Magnetic Field Laboratory (external user, Zvyagin). The UAC is particularly pleased to acknowledge the contributions of engineer Trociewitz. This is an eminent, research-enabling step forward, which shows that the EMR program has capabilities to develop unique, state-of-the-art probes.

DNP:

- The gyrotron source was modified to enable DNP experiments for both solution and solid state samples, simultaneously (spearheaded by postdoc Dubroca). This shows that the EMR program is crucial and central for the success of the DNP program. It should be noted that much of the DNP development was financed by a NSF MRI grant to PI Hill.

Recommendations – Instrumentation

1. *Make HiPER W-band EMR spectrometer available for routine use.*

DEER is a cutting-edge and effective technique in structural biophysics that should be exploited fully by using HiPER.

- Now that the high-power features of HiPER have been installed, the UAC urges that the outstanding pulse-programming software issues be solved by the St. Andrews group, so that the spectrometer can be brought into regular and routine use by users.
- Results from the first-run set of high-power HiPER experiments should be published as soon as possible, in order to showcase the capabilities and to recruit users.

- The liquid state probe head that has been developed for HiPER should be made available for routine use by users.

2. *Integrate photoexcitation.*
- Integrate ultraviolet (UV)/visible photoexcitation into the HF pulsed quasioptical EMR spectrometer, the HF cw spectrometer, and the HiPER spectrometer. A pulsed YAG laser is present on-site, and has already been used on the HF quasioptical pulsed EMR spectrometer. This will enable high-impact applications to, for example, time-resolved spin dynamics [inter-system crossing (ISC)] and photonics, electron transfer in semiconductors, solar cells, quantum computing, and magnetoreception in birds.
- Engage Engineer Trociewitz to design and lead construction of the UV/visible photoexcitation into the pulsed EPR experiments.
- Add a tunable laser source (Nd:YAG-pumped OPO), which will create a worldwide-unique user facility for time-resolved investigations of photonic materials.

3. *Extend source frequency range available for 17 T and 35 T magnets.*
- Extend to ≥1 THz the multi-frequency capability of the 17 T CW EPR spectrometer. A tunable or broad-band THz source will significantly expand the science that is accessible with the current magnet. The source, from Virginia Diodes Inc. (VDI), should also cover gaps in the current source frequency range. The terahertz source is acutely needed to facilitate full utilization, and provides world EMR leadership opportunity, which will come on-line with the series connected hybrid (SCH) 35 T magnetic field.
- Extend sample temperature capability of the 17 T CW EPR down to 300 mK. Benefits include the study of Science-Driver topics in magnetically ordered ground states and spin frustration at $T \leq 2$ K, using temperature as a third axis.

4. *Expand capabilities of quasioptical pulsed EMR spectrometer.*
- Purchase a high-power source for the quasi-optical pulsed-EMR spectrometer, to improve SNR and to increase the range of timescales that can be accessed. This is required to maintain world leadership by enabling >220 GHz operation (specifically, 330 GHz).
- Introduce phase-quadrature detection.

Recommendations – Personnel, Other

1. *Spread staff expertise*.
At least two permanent/scientific staff members should be expert operators of each of the following instruments: HiPER, 17 T transmission CW EMR spectrometer, pulsed quasioptical pulsed EMR spectrometer. Implementation of this plan will make the group more robust to unforeseen personnel changes.

2. *Get a stable hire for an EMR administrative assistant position*.
Hire an administrative assistant for the EMR program. Organization and book-keeping is severely compromised by the lack of stability at this position. This affects user administration, user statistics, UAC committee terms and elections process. Reliable administrative support is absolutely critical for the well-being of the EMR users program.

3. *Organize EMR user workshop.*
Convene a regular EMR user workshop to continue to build and broaden the EMR userbase, and to continue to expand the science output of the EMR program. The user workshop should be held annually at the NHMFL.

III. ICR

ICR UAC and contributors to this section of the report:

At UCM: Jon Amster (Chair)
Michael A. Freitas
Forest White
Elizabeth Kujawinski.

Remote participants:
John Shaw
Michael Chalmers

Summary

The ICR User Advisory committee was very impressed with the progress of the diverse set of projects ongoing at the ICR User Facility. The NHMFL ICR Facility continues to maintain its competitive edge with regard to the innovations in ICR magnet and MS instrument design. The UAC was especially impressed by progress implementing the high field 21T horizontal bore, zero boil-off magnet. The facility has continued to advance innovations in ICR cells and instrument carts.

The facility has actively pursued the application of this instrument to a variety of applications. Successful application of ICR to the challenging field of petroleomics has opened many avenues for analysis of other complex analytes. Several examples of emerging scientific applications were presented that included natural organic matter, lipidomics, metabolomics and clusters/nanomaterials. The UAC felt that there is potential for growth in these areas by leveraging the tremendous domain knowledge borrowed from the field of petroleomics.

The ICR facility is also considered a leader in the application of ICR to Hydrogen Deuterium eXchange (HDX). High-field ICR enables structural analysis of larger protein complexes. The UAC supports the application of ICR to the emerging bioanalytical challenges in the fields of lipidomics, metabolomics, top-down proteomics, native MS and MALDI imaging. The UAC feels the hire of the biological applications director staff position will be critical for application of high field ICR to biological problems.

Petroleomics

- This application is a clear scientific and economic driver for the development of higher field magnets, improvements in cell design and innovations in the ICR technique.
- The ICR has done a tremendous job developing novel methods for complex mixture analysis. The UAC supports the facilities decision to transition from method development to greater emphasis on implementing methods for large scale analysis of samples. Separations are still an important component of complex mixture analysis. The UAC

supports continued development of offline and online separations techniques for all applications.

- The UAC was impressed with the efforts to develop industrial collaborations and submit joint industrial proposals to support research activities.
- The UAC was also impressed with the development of chemometric tools for petroleomics. The facility has taken the UACs advice to distribute the PetroOrg software to users at no cost. Continued improvement and extension of software for analysis of complex mixtures whether in petroleum or other types of mixtures is fully supported.
- Ion Mobility Separations were proposed as a method for separating isobaric isomers in complex mixtures, with potential application to a variety of ongoing research.
- PetroOrg has been modified to work with IMS data as well as high res ICR data.

Natural Organic Matter

- Existing capabilities address current user needs very well.
- Program is well-administered by a new and energetic staff scientist. Program development shows promise for growing user base in this area.
- NOM-specific data processing and visualization software (EnviroOrg E-1.0) is under development, building on previously successful algorithms in PetroOrg. We recommend that this software be made available (free of charge) to all environmental users.
- Analysis of environmental mixtures could be a growth area for the science capabilities at the facility. Application of separation and ionization techniques proven to be successful in petroleomics could be applied here to complement existing capabilities and develop new science areas in NOM characterization and environmental processing.
- Cross-over science questions between petroleomics and NOM would be a particularly ripe area for growth for the facility. These questions could include fracking, oil weathering and pollution, and combustion-derived compounds in different environments.

Carbon Clusters

- The laboratory has built a state-of-the-art laser-desorption/supersonic-expansion cluster source that allows them to prepare metal clusters and organometallic materials, such as metalloendofullerenes.
- This instrumentation will be useful for nano-materials research and discovery of new nano organometallic materials and fundamentals of new materials synthesis.
- This application can establish links with the medical and astrochemistry research communities.
- The laboratory is engaging scientists from academia and government laboratories; Seven nanomaterials collaborators/users and five astrochemists/astrophysics users have been identified (including NASA).
- The high-resolution of FTICR provides the means to identify the stoichiometry some of novel clusters.

HDX, Metabolomics, Lipidomics

- Metabolomics is an exciting growth area where the facility's existing expertise in complex mixture analysis could be successfully leveraged. The lab is already moving in the direction of LC/NMR/MS through collaboration between the ICR facility and the

NMR facility. We encourage continued collaboration with experts in LC-NMR, such as Rafael Brüschweiler.

- In particular, building a focused capability in LC-based untargeted metabolomics could be a core strength in the next 2-5 years, particularly in the area of metabolite identification and integration into the biomedical sciences.
- Developments in lipidomics are moving apace and we encourage the facility staff to continue these developments.
- HDX experiments continue to be a strength of the current facility, with capabilities that are uniquely available to this facility's users, for example using resolution of isotopic fine structure to circumvent the conventional methods of deconvolution of overlapping isotope distributions.

Top Down Proteomics

- Top-down mass spectrometry of large proteins continues to be a driver for high-field FT-ICR MS instrument development
- Significant improvement in LC-MS/MS at 21T will drive demand from top-down MS of proteins over 30 kDa.
- Existing collaboration with Neil Kelleher's MS center at Northwestern has brought state-of-the art top-down proteomics methods to the 21T. This will greatly benefit the user community.
- Separation of proteins remains a challenge for top-down LC-MS/MS. Continued development in this area is justified.
- The apparent mismatch between product ion spectral complexity and the product ions assigned with the current top down database search software could become a critical issue if data obtained with the 21T system cannot be presented to the search engine. Innovation and improvements in top-down software development are needed.

Imaging, Native MS

- Small molecule tissue imaging of drugs, metabolites, and peptides is a clear driver for high resolution and mass accuracy. 21T instrument will allow for faster analysis time and improved analysis of selected molecules. This application area has potential for high impact. The ICR facility should prioritize development of an imaging source in the near future and should engage with leaders in this area.

Software

- We recommend consideration of implementing applications as server-based or Software As a Service.
- We recommend implementation of open-source data formats for integration with emerging data repositories for complex mixtures (e.g. MetaboLights).

ICR User Facility

- The ICR Facility has an impressive number of users / PIs and has been growing nicely over the past five years. In 2015, the total number of users/PIs is expected to be the highest since 2011.

- According to surveys, users are very satisfied with the ICR program, its capabilities and its staff. One continued challenge is the user response rate. Options for improved surveys or different survey times are being discussed.
- Only a few additional requests were made by users including additional options for advanced top-down capabilities, additional fragmentation capabilities and additional separations for complex mixtures. The ICR Facility has plans to address each of these requests.
- Outreach programs have been successfully implemented for K-12 students.
- Undergraduate research opportunities are available.
- ICR Program participates in MagLab Summer School
- We recommend an ICR Specific Summer School with external instructors and engagement from the UAC.

Instrumentation

- UAC was impressed with the progress on bringing the 21T online. The instrument is performing extremely well.
- Developed a novel ICR cell for $3\,\Omega$ detection. The progress is exceptional, but improvement in sensitivity is still required. The UAC supports continued development of the 3Ω cell as well as other novel designs.
- The facility has plans to develop a vacuum system that allows coupling with multiple vendor instruments.
- The facility will begin development of an in-house frontend controlled by the in-house data acquisition software. This plan is critical for testing cart innovations
- The implementation of fETD and UVPD will provide users with greater fragmentation options. These techniques will be valuable for future offline and online top-down MS.
- Coupling IMS with ICR was discussed. The UAC supports technology innovations in this area. One of the challenges in this area is the different time scales associated with IM and ICR. Long-term option is to use IM to filter isomeric species based on drift time for analysis by ICR.

Biological Applications Staff Search

- Four applicants for the biological applications position were identified and discussed.
- The UAC supports the move to immediately interview the top three candidates.

To do Items for the UAC

The UAC needs to reconcile terms with the MagLab committee bylaws. Two members need to rotate off early, and two members need to rotate off late.