**Report on the 2014 NHMFL User Advisory Committee meeting**
**Held in Tallahassee, FL, from October 10th to 11th, 2014**

**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: James Analytis (University of California, Berkeley), Kenneth Burch (Boston College), Jason Cooley (Los Alamos National Laboratory), Nicholas Curro (University of California, Davis), Ian Fisher (Stanford University), Nathanael Fortune (Smith College, Executive Committee Member), Madalina Furis (Chair for DC/ Pulsed Field /High B/T, University of Vermont), Jeanie Lau (University of Calfornia, Riverside), Wei Pan (Sandia National Laboratory), Cedomir Petrovic (Brookhaven National Laboratory**)**, Makariy Tanatar (US DOE The Ames Laboratory), Chris Wiebe (University of Winnipeg, User Committee Chair)

NMR/MRI committee: Robert 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), Len Mueller (UC Riverside), Fang Tian (Penn State University), Tatyana Polenova (University of Delaware), Scott Prosser (University of Toronto), Linda Columbus (Virginia)

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.), Sergei Zvyagin (Dresden High Magnetic Field Laboratory, Helmholtz-Zentrum Dresden-Rossendorf, Germany)

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)

Committee members who served in 2014 and are now retiring: Ian Fisher, Nick Curro, Cedomir Petrovic, Dmitri Artemov, Ari Borthakur, Conggang Li, Mark Rance, Ivan Tkac, and Joshua Telser

On behalf of the User Committee, we would like to express our appreciation for the NHMFL and Florida State University for hosting a productive and well-organized meeting in Tallahassee for all three branches of the magnet lab. The feedback from the attendees was enormously positive, and many of the queries that arose prior to and during the meeting were addressed in a timely and efficient matter.

The National High Magnetic Field Laboratory, consisting of three separate branches in Tallahassee, FL, Gainesville, FL and Los Alamos, NM, continues to set the pace for cutting-edge research in high magnetic fields across many sub-disciplines of science and engineering, including condensed matter physics, solid-state chemistry, medical imaging research, and the design of state-of-the-art instrumentation. By nearly every metric, the NHMFL shows continued growth in terms of users, scientific publications, world-records in magnetic field production, and educational outreach. We are optimistic about a very bright future for the NHMFL, and wish to thank the three facilities and their affiliated host institutions (Florida State University, the University of Florida, and the Los Alamos National Laboratory) for their continued leadership, vision, and support.

## (1) Executive summary

Many of the topics discussed at the User Committee Meeting had a broad impact over the entire user base, and these will be discussed first before moving on to particular topics of the sub-committees concerning their disciplines. The overall discussion of these topics is ongoing, but productive, and many of the views here represent the majority of the represented community.

### (i) Focus of the magnet lab

The User Committee gives a strong endorsement for the current vision and allocation of developmental priorities at the NHMFL. Recent choices of resources, in terms of investments in key magnet technologies, personnel, and future scientific endeavors, are all widely accepted and supported by the general user committee. Many of these particular choices will be outlined in the sub-committee reports below, but our overall assessment of the priorities set by administrative staff at the magnet lab are overwhelmingly positive, and the user base feels that most of its needs are met and usually exceeded.

### (ii) Housing

The issue of housing at the Tallahassee branch in particular continues to be a concern for users. While there has been some positive developments with the lack of quality housing for visitors to the magnet lab, we feel as a community that future development is needed, especially with the growth that the NHMFL as seen in users. We strongly endorse for Florida State University to continue to work with the National High Magnetic Field Laboratory to work on bringing a guesthouse to Innovation Park for visiting users. For a variety of reasons, including personal safety and fast accessibility for users, we feel that it is crucial for concrete steps to be taken to bring a long-term plan for housing close to the Tallahassee lab closer to a reality.

### (iii) Future hiring strategies

(a) The NHMFL has truly excelled in hiring the very best scientists and engineers over the years. The unique resources at the lab (including personnel, facilities, and access to new techniques), continues to be a great draw in attracting world-class talent. We encourage the NHMFL to continue this standard of excellence and to include the User Committee in the hiring process of new talent to the magnet lab, and we especially look forward to increasing the numbers of senior staff members. Many of our suggested future hires are listed in the sub-committee sections below.

(b) The User Committee is very encouraged by recent developments by FSU to secure a faculty line for a senior hire that would fill the role of Chief Science Officer for Condensed Matter Physics. This is increasingly important for the continued vitality of the laboratory and for the recompetition process. We encourage the NHMFL to draw upon the resources of the UAC and members of the scientific staff throughout this process.

### (iv) Diversity

The User Committee is continually impressed by the efforts of all three branches of the NHMFL to increase diversity. The UAC was struck by the observation this year that our committee, itself, needs to increase its diversity. We would like to make a recommendation for the lab and current members of the UAC to choose a more diverse pool of nominees for future members of the UAC. The UAC is also a resource that the NHMFL can use to draw upon in the future for recommendations of hires that would increase diversity.

### (v) The UCGP

The review of the UCGP was timely, thorough, and very informative for the UAC. The general view of the UAC was that this program is a success at the NHMFL that seeds new initiatives, establishes new young scientists, and brings new techniques up to the level that they can transition to the user program. It is not immediately clear if or how the UAC might be helpful in the process, but there was a general sentiment that there is at least a potential for beneficial interaction, possibly in the realm of providing additional perspective that might be helpful in forming an assessment of the relative merits of proposals (for example, the eventual user impact). Equally, there was also a general consensus that it is useful to have some input from upper administration for the allocation of funds within the current needs of the NHMFL community.

**(vi) Safety**

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**

The UAC applauds the highly successful summer schools organized by the NHMFL which has a tremendous impact on new and future users. This program is truly inspirational and has a overwhelmingly positive effect on future researchers, and on the staff involved.

**(viii) User committee changes**

(a) The UAC briefly discussed regularizing the Bylaws to be the same process for each of the divisions. The current suggestion is to modify so that the timing of nominations and elections, and the tenure on the committee (3 years) is the same for all divisions. We look forward to the recommendations of the NHMFL regarding these changes.

(b) Changes to the executive committee: The following new positions have been established by the User Committee:

NMR/AMRIS (2): Linda Columbus, Rob Schurko
EMR (1): Kurt Warncke
ICR (1): Jonathan Amster
DC/PFF/High B/T (3): Chris Wiebe, Madalina Furis, Nathanael Fortune

## (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:

**James Analytis** (University of California, Berkeley)
**Kenneth Burch** (Boston College)
**Jason Cooley** (Los Alamos National Laboratory)
**Nicholas Curro** (University of California, Davis)
**Ian Fisher** (Stanford University)
**Nathanael Fortune** (Smith College, Executive Committee Member)
**Madalina Furis** (Univ. of Vermont; vice-chair for DC/ Pulsed Field /High B/T)
**Jeanie Lau** (University of Calfornia, Riverside)
**Wei Pan** (Sandia National Laboratory)
**Cedomir Petrovic** (Brookhaven National Laboratory)
**Makariy Tanatar** (US DOE The Ames Laboratory)
**Chris Wiebe** (University of Winnipeg, User Committee Chair)

(i) **General comments**:

The user committee commends the DC/Pulsed Field/ High B/T facilities for the outstanding support offered to users and continuing efforts to remain at the forefront of magnet science. We continue to enthusiastically support the plans for a high $T_C$ superconducting magnet and reiterate our excitement to the definite progress on a 40 T / 28MW resistive magnet development. We find that a key priority for the lab should be a second DC system that gives access to fields up to 40 T (the hybrid is heavily oversubscribed; several of the experiments that require the hybrid could be accommodated on the 40 T resistive magnet). Furthermore, if the hybrid suffers either a short term failure, or a longer term problem, there is a second magnet that would be able to serve users who need fields in excess of 35 T. This would also help the magnet lab maintain its competitiveness with respect to the other DC field labs.

The user community is very happy with the new 65 T pulsed magnet coil design that enables longer pulses and faster cooldown as well as the undergoing efforts for the 100 T redesign. There is a general consensus the new magnet engineer hire is a great success for the PF facility and the Maglab in general. Doan is dynamic and he already has plans for a 105 T magnet, using a smaller 7mm bore. Plans for a duplex magnet based on firing two capacitor banks are also exciting. We also note the plan for a cryostat with a space-free –optics compatible tail that would operate in a horizontal geometry. While this may not be a priority for the coming year, we encourage the lab to continue exploring this idea.

The committee endorses the idea of an 18 T/Dilution Refrigerator system for the High B/T facility. As an External Advisory Committee recommendation, this system would remove considerable pressure off the user schedule at this facility. While the design would be somewhat similar to some systems in the mK facility, the 18 T would be used for longer experiments than typical mK runs at the Tallahassee facility. It would also give users a fast turn-around system, which they are currently lacking.

On the experimental techniques development front, we strongly endorse attempts to increase the signal-to-noise ratio for the DC lab experiments. The commitment to continually striving to improve experimental techniques in parallel with development of new magnet technologies is a real strength of the DC program.

There was a general consensus that accepting common proposals for the DC and PFF facilities is very beneficial for users and experiments that need to explore a large parameter space in pursuit of one science goal (especially where different approaches of the same experimental techniques are better suited for different regions in that parameter space). The flexibility of collaborating with the same staff scientist at both facilities is, in this context, much preferred and appreciated.

The committee members discussed at length the proposal to split the DC and PFF committees. The consensus was that separating the DC and PFF committees would possibly bring more needed PFF expertise to the user committee as a whole. A final decision in favor of forming a separate PFF committee should come with provisions towards keeping the communications lines open with the DC committee to protect the interests of users of both facilities. By and large, the positives of splitting the committee outweigh the negatives and the committee feels synergies between the two programs can still be maintained.

**(ii) <u>The DC/Pulsed Field Optics Programs Review</u>:**

The user committee requested that the DC and Pulsed Field facilities provide a detailed status report of the optics programs, the first one since the 2009 Workshop on Optics in the Florida Split Helix Magnet that set some directions for the future development of the optics program, on the eve of commissioning the first 25 T split-coil magnet compatible with free-space optics techniques. We summarize below our conclusions and feedback from users.

First and foremost, the committee is unanimously applauding the outstanding progress at both facilities. The committee recognizes the tremendous progress in the development of multiple, cutting-edge techniques, in particular ultrafast visible/near IR optics and Raman. This progress is in part due to the introduction of the split Helix magnet which attracts the interest of a wide array of new users. User support scientists have done a fabulous job of setting up the next generation of optics in high magnetic field experiments in the Helix and the pulsed field facility.

Specific achievements the committee wishes to point out for the optics program include:

- Ultrafast polarization and time-resolved techniques using a state of the art, multifunctional new laser amplifier system that cover a broad spectral range with instrument-limited temporal resolution. (Seed: 10nJ, 80MHz Amplifier Pulse energy 5mJ, repetition rate 1 kHz, pulse duration 15 fs OPA -250nm-10μm pulse duration approx. 60-80fs)
- Ultrafast two-color pump probe
- Magnetic Circular Dichroism in thin film and solution (S/N approx. 0.0001)
- Capability for high intensity THz Generation (See laser spec above)
- 2D Coherent Ultrafast spectroscopy
- Spatially –resolved Raman spectroscopy and T< 1K Raman
- Mid IR single turn (150T+) cyclotron resonance

Most of the instrumentation and techniques have never been used in magnetic fields larger than 9 T prior to these developments.

The user community also appreciates the large diversity of optical spectroscopy experiments developed. Unlike other experimental techniques, spectroscopy involves a larger degree of customization added to the "standard" experiments list and the users would like to see that continues in the future. While better synergy between the various facets of the optics program (i.e., visible, far infrared, Raman and pulsed fields) should be improved, users don't want to see that happening at the expense of limiting or cutting the great experimental portfolio that is so essential for the success of the optics program. They do recognize that a senior, full professor level hire in optics and spectroscopy might help this synergy, and they caution it is critically important that this future leader is someone with a commitment to a broad and inclusive vision of this field and appreciative of the great diversity of science enabled through optical experiments.

The program is now ready to move to the next level where many of these experiments that are unique need to be streamlined in order to serve a larger user base (i.e., spatially-resolved Raman, most Helix experiments, etc.). Given the diversity of techniques that now exists, the committee believes DC facility optics would greatly benefit from an additional dedicated technician that could be trained in standard laser maintenance procedures and daily optics setup debugging. Staff scientists in optics are seriously overworked to the point of compromising their own in-house research program, and thus the development of future advances.

In terms of further infrastructure development, the HELIX has clearly enabled a large array of new experiments, but the magnet geometry is really not ideal for most of the Faraday geometry experiments. While the range of experiments available at the DC facility is quite impressive, the users also think that the community could benefit greatly from the introduction of FTIR techniques into the Split Helix and ***they strongly emphasize the absolute need for the Faraday insert that would enable the Faraday geometry necessary for about 90% of ALL experiments in the HELIX***.

We believe that the pulsed facility could greatly benefit from a similarly optimized magnet to further expand the range of optics. The user committee is grateful to see that the PFF is considering such a design, though we suggest they consider a split-coil type system and encourage them to reach out to a wide array of potential users who could help in thinking through the design and potential experiments. There is particular interest in developing the ability to do initial experiments at the DC facility that can be extended to the pulsed facility as well as broadening the spectral range available at the pulsed facility.

The users understand that perhaps the issue of reaching base temperature as low as 5 K in the Helix cryostat may be challenging, but the committees wishes the Maglab to continue exploring that idea, since the 5 to 15 K range is crucial to many optics experiments.

### (iv) Priority list of recommendations and executive summary:

- Management should support the hiring of a FCE position to assist with the development of the optics program.
- We endorse the hiring of a new physics faculty member at FSU who can assume the role of CSO in experimental condensed matter at the NHMFL.
- We recommend the continued development of new DC magnets, and in particular the 40 T/28 MW resistive magnet. The hybrid magnet is currently oversubscribed, and the 40 T would greatly relieve some of the stress on this facility. In addition, if the hybrid magnet fails, the 40 T would serve as a back-up system so that high field science can continue to flourish without a significant impact to the user program.
- Increased synergy between the DC and PFF is strongly endorsed for the future, even though we are considering separating the user groups to address specific concerns of each facility.
- We recommend future developments of higher fields at the PFF and new techniques. Plans for a duplex magnet based on firing two capacitor banks are exciting. Pulsed NMR experiments might be explored.
- We recommend looking into the possibility for a 18 T/DR magnet at the high B/T facility. This could be used for experiments which are longer in time scales compared to experiments at the Tallahassee branch, but shorter than the experiments currently completed in Gainesville. This would enable a faster turn-around for the high B/T facility in general.
- We strongly encourage the continued repurposing of older magnets, which is an extremely cost effective way of building high field instruments at the MagLab (this is also endorsed by the MR report below).
- We note the value of the UCGP program at this point, and encourage its continuation and expansion, as it has provided funds to develop hardware and support a number of other worthwhile projects and research areas (this is also endorsed by the MR report below).
- The UC is concerned that developments in high field magnet technology would be dramatically compromised by a change of site, and see no conceivable reason that would justify disrupting the current situation (this is also endorsed by the MR report below).

## (3) Report of the Magnetic Resonance Division User Committees

**Sections: I. NMR and MRI, II. EPR and III. ICR**

### I. NMR and MRI

**NMR/MRI UC and contributors to this section of the report:** R.W. Schurko (Chair, Windsor), M. Pruski (Ames Lab, Iowa), Michael Harrington (Huntington Medical Research Institute), Brian Hansen (Aarhus), Eduard Chekmenev (Vanderbilt), Len Mueller (UC Riverside), Fang Tian (Penn State), Tatyana Polenova (Delaware), Scott Prosser (Toronto)

**(i) Overview:** The NMR UC is very pleased with the progress made in the NMR/MRI division over the past year *across the board*, including publication count and quality, user recruitment and activities, development and application of new experimental methods, applied projects in materials science, structural biology and MR imaging, and magnetic field technology development. Continued investments in personnel/support staff, faculty, equipment and facilities are all strongly endorsed. The NMR/MRI division continues to make the MagLab one of the premier sites in the world to do groundbreaking experiments in NMR.

**(ii) Personnel**: We are very impressed with the high quality of intellectual engagement and scientific leadership of Prof. Lucio Frydman in several major project areas, including the development of DNP methods and applications of MRI. Aside from spending 9+ weeks on site per year, he is in contact (via Skype, etc.) with Florida-based team members on a weekly basis. Productivity in terms of research progress and publication output is also impressive. We are also very glad to hear of the hiring of Dr. Yan-Yan Hu by the Department of Chemistry at FSU; her expertise in battery materials, interest in biomimetic composite materials, and proposed use of the DNP NMR facilities for these projects makes a crucial link between her department and the NHMFL. Earlier in the year, we were disappointed to hear of the departure of Prof. Rafael Bruschweiler, a world-renowned expert in NMR, from the Department of Chemistry at FSU. We strongly endorse the hiring of a new NMR faculty member at FSU, who can assume the role of associate director of biophysics (or another strategic area) at the NHMFL. We were pleased to hear that UF is considering other faculty hires to better utilize the AMRIS facility (e.g., brain research, metabolomics). We have several staffing concerns in the NMR area at the MagLab; in particular, we strongly suggest the hiring of two FTE positions: one for SCH/HTS magnet/spectrometer development and one for DNP applications and development. An enormous investment has already been made in these areas in terms of infrastructure – if they are to truly flourish, additional personnel must be put in place, with the eventual goal of translating them to user support and research personnel. Finally, management should explore the cost effectiveness of hiring a ½ FTE position that can deal with repairs of equipment (e.g., rf, amplifiers, etc.) that would otherwise cost too much to replace/repair via commercial sources.

**(iii) Equipment, Infrastructure and Use of Facilities:** Between the MagLab and AMRIS, there are 18 NMR and MRI spectrometers and magnets, with fields ranging from 3.0 to 21.1 T, which are used in a diverse range of applications including SSNMR of materials and biosolids, solution NMR (e.g., proteins, metabolomics, clinical) and imaging of animals and humans; this includes spectrometers incorporating new and innovative technologies such as DNP (dissolution, solutions and solids), HTS magnets, cryoprobes, and specialty probes developed at the MagLab (for solids, imaging, etc.). The spectrometers range in installation dates from 1994-2013, and several recent upgrades have been made. We strongly recommend the provision of both resources and support necessary to maintain and effectively use this impressive fleet of NMR spectrometers – failure to do so would undermine many of the ongoing projects, as well as the investments in this equipment.

*Highlights.* At FSU, the 900 MHz spectrometer, which has been active for 10 years, is the current flagship instrument of the MagLab. Its time is split between the SSNMR user program and MRI (in-house probes, mainly small animals). The 830 MHz narrow-bore magnet is used for materials SSNMR, and has freed up much time on the 900 MHz. The same can be said for the 800 MHz/63 mm system, which is a repurposed magnet from UMinn. Commercial NMR electronics and an in-house 3.2 mm MAS HXY probe (built by Gor'kov) have allowed increased materials and biosolids NMR experimentation. We strongly encourage the continued repurposing of existing magnets, which is a cost-effective way of building high-field instruments at the MagLab. In particular, we support the idea of acquiring another 800 MHz magnet, which will feature a low-E 3.2 mm probe and be coupled to a Bruker HD console. The UCGP (vide infra) has provided funds for the development of a 100 kHz MAS probe for this system.

At AMRIS, there has been much progress with DNP and imaging. The dissolution DNP NMR spectrometer (1 K/5 T/212 MHz/140 GHz) is being applied for in vivo metabolism studies, and will permit imaging of low-gamma nuclides; signal enhancements of 16000 and 11000 have been reported for 4.7 and 11.1 T, respectively. Future visions include a users facility for continued and future use in in vivo, multinuclear NMR, MRI and metabolomics projects. There is progress in the continued use and set-up of the solution and solids DNP NMR spectrometer, which will incorporate a single gyrotron and two 14.1 T magnets. Furthermore, continued projects in MRI continue successfully at AMRIS (NMR, clinical and animal/human imaging).

*Publications & Funding.* In 2013, publication output and funding for both locations is also very impressive. The FSU team reports 43 publications (11 in high impact journals), 210 users and 2775 user days on 9 instruments. AMRIS reports 51 publications, 184 users, and 1000+ users days on 8 instruments. We note the value of the UCGP at this point, and encourage its continuation and expansion, as it has provided funds to develop hardware and support a number of other worthwhile projects and research areas.

*User recruitment and outreach.* User numbers are good, but continued efforts must be made to increase the user base at FSU and AMRIS. In 2013, workshops were held which discussed potential use of SCH magnet spectrometers (annual UCM) and coil building for MRI (AMRIS). A 2015 workshop on high field magnet technology is currently being organized – we believe more such workshops are necessary. The NMR UC is delighted with the format of the new web site, and believes it will play a major role in attracting new users, collaborators and public interest, and should also serve to streamline the proposal review process.

**(iv) Magnet Technologies**: One aspect of the MagLab which differentiates it from all other facilities in the world is the continued development of new magnetic field technologies that will undoubtedly 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 – some never attempted before by NMR spectroscopists. We will focus our discussion on the latter three systems for the purposes of this report.

The ***36T SCH system*** can ramp from 0 to 36 T in 15 minutes, has a field homogeneity of 1 ppm and no long term drift, and will be commissioned in early 2016. The committee is impressed by and strongly endorses the development of SCH probes, including low gamma MAS, CP MAS, static (PISEMA), static low gamma HX, a Zilm 1.6-mm $^{1}$H-detection MAS probe and an imaging probe. An HR-MAS probe is being developed as a test instrument, and pending future improvements in resolution, the development of an HXY MAS probe for biosolids is a high priority. Static broadband probes are a very high priority, since sub-1 ppm resolution is unnecessary, and can enable multinuclear SSNMR studies of many new materials. The proposed development of the ***30 T NMR system*** using HTS magnets is very exciting. New materials such as the REBCO tape and Bi-2212 round wire HTSs are being compared, and a demonstration magnet (Platypus) is in progress, with ca. 24 T as an intermediate goal. HTS technologies continue to be developed and advance, but resolution on the ***45 T Hybrid*** is still ca. 100 ppm (the SCH system can act as an intermediary system during development). Projects on increasing field homogeneity, probe design, power supplies, etc. are either in progress or will be initiated.

Support for these projects, in terms of funding and personnel, is absolutely essential. All of the NMR and MRI UC members can envision spectrometers operating at these magnetic fields which will enable new classes of experiments that will push the boundaries of what is possible, and permit the study of complex materials and biological systems that have hitherto been impossible to probe via NMR methods.

**(v) DNP**: We are very pleased with the excellent progress on the installation of the 395 GHz/600 MHZ DNP NMR instrumentation. Within the last few months the smart and cost-effective idea of coupling one gyrotron with two 14.1 T NMR magnets to serve both MAS DNP and Overhauser DNP (ODNP) instruments became a reality. The microwave (MW) beam splitter has been installed on a quasi-optics table, providing a flexible tool for the management of MW power, which can be independently controlled for both instruments. The MAS probe and associated cryogenics are already operational, but need additional adjustments of cryogenic gas delivery and minor facility upgrades (continuous source of compressed dry $N_2$ gas) to enable reliable 24/7 operation. The DNP MAS NMR capabilities should be available to users in 2015 after the installation of an NIH-funded field-swept magnet. The installation of ODNP instrument is also progressing well, with the commencement of user activities scheduled for 2017. The MAS and ODNP capabilities will complement the dissolution DNP user program at AMRIS. We note that active collaborations between the NMR and EMR divisions have made much of this possible.

The NHMFL staff recognizes that further fundamental research is needed to reach the full potential of DNP in enhancing NMR's sensitivity. They installed an innovative console-controlled gated shutter, which is able to deliver the MW irradiation in pulsed mode, and thereby permit to study transient phenomena relevant to the DNP process. We strongly recommend that funding be provided for continuation of these efforts. DNP is a very labor-intensive technique, which requires constant attention of highly qualified scientific staff. As described in the personnel section above, we recommend the initiation of a search for a FTE staff scientist to work on the DNP development and facilitation of collaborations with users.

**(vi) MRI:** The Maglab continues to house some of the world's most advanced instruments for high field MRI and MR microscopy. The personnel on site are a valuable resource to users and collaborators alike and the work from the Maglab is an inspiration for the entire imaging community. It is important that the collaboration to magnet industry (Bruker) is healthy – the Maglab should continue to be a high priority customer to Bruker. Compared to some of the large scale projects launched in recent years (e.g., human neuroimaging, Connectome project), we believe that the microimaging is a very cost effective route to improve on our ability to interpret clinical MRI through validation studies and cellular level imaging. There continues to be great potential for gaining valuable insight into MR signal formation at the cellular level, especially microstructural imaging with diffusion weighted MRI and in vivo diffusion spectroscopy. For these reasons, we are pleased the MagLab responded to recommendations from the 2013 Users Committee by developing approaches to obtain imaging capability on the new generation of high field magnets (36 T). Long-term support for the continuation (hardware and staffing) of MRI hardware development is vital for the long-term success of high-field MRI. For external users planning to do in vivo work on their own animal models, it is important that a system is in place to facilitate transfer to and housing of animals at the Maglab site.

In regard to a competitive renewal for the MagLab, we are very concerned that science in high field magnet technology would be dramatically compromised by a change of site. Extra-ordinary progress at the current sites has been achieved with a user-responsive and highly sophisticated physical, professional, educational, and staffing infrastructure that will be grossly disrupted for users by any change of the principal locations. The current users do not see any conceivable advantage that might justify disrupting this situation.

**(vii) Priority list of recommendations and executive summary.**

- Management should support the hiring of a minimum of two FTE positions: one for SCH/HTS magnet/spectrometer development and one for DNP applications and development. Part time personnel for electronics/rf repair is also recommended.
- We endorse the hiring of a new NMR faculty member at FSU (to replace Rafael Bruschweiler) who can assume the role of associate director of biophysics (or another strategic area) at the NHMFL.
- We strongly recommend the provision of both resources and support necessary to maintain and effectively use the impressive fleet of NMR spectrometers (18 in total between NHMFL and AMRIS); to pull back on this enormous investment as it is really starting to flourish would be unwise.
- Support for the HTS, SCH and hybrid magnet/spectrometer projects, in terms of funding and personnel, is absolutely essential. Within a few years, these systems will permit new NMR and MRI experimentation that was previously not possible; technologies from these developments may advance the entire field of NMR/MRI as a whole.
- We strongly recommend that funding be provided for continuation of efforts in DNP NMR. European institutes are currently far ahead of the U.S. in this area; it is crucial that new techniques and applications continue to be spurred at the NHMFL.
- We strongly encourage the continued repurposing of older magnets, which is an extremely cost effective way of building high field instruments at the MagLab. In particular, the acquisition of another 800 MHz magnet is supported.
- We note the value of the UCGP program at this point, and encourage its continuation and expansion, as it has provided funds to develop hardware and support a number of other worthwhile projects and research areas (this is also endorsed in the DC/PFF/High B/T report above).
- Efforts should be made to facilitate transfer to and housing of animals at the Maglab site.
- The NHMFL should maintain a healthy relationship with Bruker for acquisition of new equipment and perhaps future exchange of technologies and IP.
- The UC is concerned that developments in high field magnet technology would be dramatically compromised by a change of site, and see no conceivable reason that would justify disrupting the current situation (this is also endorsed in the DC/PFF/High B/T report above).
- Finally, we recommend that the Users and MagLab scientists contribute a prominent science publication for 2015 on the advances that will arise from the SCH, HTS and/or DNP systems, in order to inform the wider community prior to the rollout in 2016 of this unique instrument, and encourage proposals from external users.

## II. EPR

**EMR UAC 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.), Sergei Zvyagin (Dresden High Magnetic Field Laboratory, Helmholtz-Zentrum Dresden-Rossendorf, Germany)

### (i) Director and Personnel

We were overall very impressed with the accomplishments of Electron Magnetic Resonance (EMR) program. Our review of the research output (count and content) over the 2013-2014 year, on-site viewing of the facilities, and interaction with EMR personnel, confirms that the EMR program at the NHMFL continues to be a world-leading high-field (HF) EMR facility. The EMR program continues to be remarkably productive, with 30 papers already published in 2014. The continued increase in the EMR user base (growth from 109 to 144 users over the previous, including 19 new PIs) is impressive.

EMR Director Steve Hill provides effective leadership, and wields both in-depth technical expertise and knowledge of the science being performed by EMR group personnel and by users, which underpin a strong vision for the future of the EMR group. Hill displays a high degree of commitment and responsiveness to user needs, and user-driven scientific directions. This commitment and activity keeps the EMR user program at the forefront of high field EMR technical development and science.

EMR staff (Jurek Krzystek, Andrew Ozarowski, Likai Song, Johan van Tol, Sebastian Stoian) are all technical experts, and are driving the science in their areas with their respective instruments. New Dynamic Nuclear Polarization (DNP) –area postdoc Dubroca and EMR engineer Trociewitz, are viewed as valuable new contributors to the DNP effort. The close involvement of University of Florida (UF) faculty Alexander Angerhofer and Gail Fanucci is highly appreciated.

### (ii) Instrumentation

*HiPER Spectrometer*. HiPER is a W-band EMR spectrometer that has been purchased from the group of Graham Smith at St. Andrews University, Scotland. HiPER is a state-of-the-art, high-power, high-sensitivity instrument with a unique non-resonant sample arrangement. The EMR UAC is pleased about the progress made with the HiPER instrument over 2013-2014, which includes operation at low power in continuous-wave (cw) mode, and the development of thin-layer sample holders for solution samples. A recent experiment at room temperature performed with nitroxide samples at a concentration of 10 μM shows an impressive signal-to-noise ratio (SNR) increase of ~100, relative to the resonant probe configuration of the commercial Bruker W-band spectrometer. This was achieved with samples in collaboration with Professor Gail Fanucci (UF). This result confirms expectations for the HiPER instrument, and opens the door to a wide range of nitroxide probe and label experiments. In summary, there is clear evidence of success with HiPER, and high promise for the future.

Regarding the operation of HiPER at high power, the EMR UAC is concerned that the high-power amplifier (1 kW) has not been implemented, and that there is some uncertainty about the time for implementation. Funds have been appropriated for the full implementation of the high power capability. The amplifier itself has been on site for a while. The EMR group awaits the receipt of a laser with a silicon switch, as well as an Attocube drive. The incorporation is dependent on a site visit by the St. Andrews team. We strongly encourage the effort toward immediate implementation of the high power amplifier to the extent currently possible, by an EMR Group member. The UAC feels that the negotiations with the St. Andrews group must be brought to a close and any further delays should be avoided. The high-power mode of HiPER is crucial to the user base, especially in the structural biology field.

*DNP*. The UAC is excited by the progress in the area of DNP. We appreciate that the expertise in the lab allows combination of commercially available systems with in-house equipment, especially the gyrotron source, which has been modified to enable DNP experiments for both solution and solid-state samples, simultaneously. This is proof of the strong synergy that is available between the EMR and NMR programs. We encourage continued collaboration between the EMR and NMR programs in this direction. This synergy is rapidly moving DNP forward at the MagLab, with impact on DNP worldwide.

We note that EMR can continue to make strong contributions to the advancement of the DNP program. The implementation of quasi-optical technology in the DNP spectrometer is a direct contribution of the EMR group. EMR can investigate and optimize paramagnets (molecules and solution environments, other conditions) for the generation of DNP.

The UAC is excited by the plan to integrate an EMR spectrometer into the DNP spectrometer. We hope, however, that this will not dilute personnel effort on the other experiments, such as HiPER.

*Terahertz Sources*. We are extremely interested in extending the multi-frequency capability to the high frequency range, to above 1 THz. A tunable or broad-band THz source is highly desirable. This would significantly expand the science that is accessible with the current high magnetic fields. The tunable THz is critical for the future technology and higher fields, which will come on-line with the series connected hybrid (SCH).

*Transmission Spectrometer*. A concern is the failure of the 180 A power supply for the 17 T magnet, that is part of the broadband transmission spectrometer. The power supply is broken, and needs to be replaced. The broadband transmission spectrometer is a workhorse instrument, and is now being run using an older power supply, that does not allow access to the highest magnetic fields (for which sources are available), limiting the science which can be explored. A large amount of science is produced by this instrument, and failure of the backup power supply would be catastrophic for the program.

The UAC feels that the purchase of an additional VDI source to cover gaps in the current source frequency range (approximately $60K) is necessary. This additional source will substantially improve the frequency coverage and improve data quality. A direct outcome will be to eliminate ambiguity in the analysis of experimental data.

*Quasi-optical Spectrometer*. The UAC recommends the purchase of a high-power source for the high-power quasi-optical spectrometer. This will provide the power required to improve SNR, increase the range of timescales which can be accessed, and allow for quadrature detection.

*Sample Control*. There is a view that additional sample control, excitation and detection capabilities could be expanded. For example, optical and electrical detection of EMR would broaden the scope of experiments which can be undertaken with the spectrometers, and provide access to new areas of scientific inquiry.

One area in which rapid progress in this direction could be made would be the provision of a tunable optical source. Users have requested this capability, and the UAC supports this request.

This capability would significantly expand the ability of the EMR program to contribute to the energy and environmental systems areas. The tunable laser source will also be applied to the HiPER spectrometer, creating a worldwide unique user facility for time-resolved investigations.

## (iii) EMR User Program

The user program currently operates at an oversubscription of 113%. Facility and personnel are at or beyond capacity. Given the anticipated growth in demand when the high-power capability of HiPER comes on-line, additional staff will be required to maintain user satisfaction, and the volume and quality of the user program.

The UAC supports the plan to hold an EMR user workshop by the end of Fall 2015. We also recommend that the EMR user workshop be held annually. These workshops will help to recruit new users, promote awareness among researchers in the field of the experimental capabilities at the NHMFL, and consequently further expand the science output of the EMR group.

**(iv) EMR Group Personnel**

We are impressed with the depth of expertise, and superior level of commitment of each EMR Group member. The group drives both technical innovation and the science.

There is a concern within the UAC that knowledge is concentrated in individuals, and knowledge needs to be transferred across the group. The group needs to be robust to unforeseen personnel changes. The ability of the EMR group to maintain their high-field EMR forefront position is vulnerable to this. This issue is acute in the case of HiPER, where the upcoming implementation of high power operation will require new skills and knowledge. We recommend the dedication of a group member to the implementation of high power operation of the HiPER instrument, and the subsequent management of users for this device.

**(v) Priority list of recommendations and executive summary:**

• HiPER spectrometer: Finalize implementation of high-power capability of HiPER, and make it available to the user community. If necessary, emphasize or shift personnel commitments of one group member to manage the effort on HiPER.

• Transmission spectrometer: Replace the broken power supply for the 17 T magnet. Purchase and install a new VDI source.

• Quasi-optical spectrometer: Purchase and install new source.

• Multi-instrument use: Obtain a tunable laser source for photoexcitation of samples.

• Extend the frequency range of sources to above 1 THz. This is an acute need for the Series Connected Hybrid (SCH) development.

• HiPER hire: Fill the postdoc position for the HiPER spectrometer that is currently in open-search/interview phase.

• New hire: Given the growth in the user program, and profound additional growth anticipated in the HiPER program, the UAC recommends an additional MagLab staff position within EMR. This position would expand the experimental effort in the biology area, and provide the capability to manage issues related to expertise concentration.

**III. ICR**

**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)

**(i) Summary**

The ICR User Advisor committee was very impressed with the quality of the ICR User Facility. The NHMFL ICR Facility continues to maintain its competitive edge with regard to the latest in ICR magnet and MS instrument design. The UAC was especially impressed by the high field 21T horizontal bore, zero boil-off magnet. The facility has also been active in advancing innovation in ICR cells and instrument carts. We were especially impressed with improvements in the dynamically harmonized cell and modular hybrid instrument carts that have the potential to interface with a variety of commercial MS instruments. The application of ICR instrumentation to energy/environmental applications is a unique strength of the ICR program. The UAC felt that there is tremendous potential for growth in the area of petroleomics and environmental applications.

**(ii) Instrumentation**

- The 21 T is the most important recent instrumentation development. The 21 T provides 1.1 mDa resolution up to 1.6 KDa and 3.4 mDa out to 2 KDa for complex mixture analysis.
- The 21 T was installed in June 2014 and has been running continuously at field since that time.
- ICR program continues to innovate in ICR instrument design. They have designed and built a hybrid instrument for the 21 T based on the Thermo Velos Pro with Front ETD. The Velos Pro hybrid will provide FETD, HCD and CID external MS/MS experiments. The coupling of the Velos Pro with the ICR is modular so that other hybrid configurations can be used in the future.
- New cell designs based on the Dynamically Harmonized Cell hold the potential to greatly improve ICR performance at higher field.
- UAC advises that the facility identify high-impact problems to illustrate the power of the 21 T.

**(iii) Overview of Applications**

*Biological*

- Neil Kelleher presented current capabilities and future applications of the 21 T magnet for top-down proteomics. Dr. Kelleher has a strong continuing relationship with the ICR program. He is providing the scientific leadership in developing biological applications of ICR-based LC-MS/MS for top-down proteomics.
- Top-down proteomics quantitation requires isotopic resolution. A 21 T uniquely provides this resolution from 30 KDa to 60 KDa. With regard to clinical proteomics, identification of proteoform difference can be done by top-down

proteomics. The NHMFL is the only place that would allow for the demonstration of the benefit of high-field top-down proteomics using the highly innovative 21 T. The more comparable Orbitrap instruments do not have the resolving power to breach the 30 KDa barrier at this time. Kelleher reported that for 0-30 KDa the top-down proteomics method is ready to deploy for users in a more robust application platform.

- The UAC recommended that a novel approach to perform parallel 14.5/21 T LC-MS/MS analysis be considered as an opportunity to showcase the capabilities of both instruments in this growth area.
- An immediate concern of the UAC is the appointment of a biological-applications director to facilitate biological applications of ICR. The bio director should bring a vision of how ICR (not just the 21 T) intersects with bio driven applications to fully capitalize on the strength of ICR.
- Potential areas for additional bio-applications include metabolomics and small molecule imaging. Large protein structure/proteoform/PTM mapping would be a strong application, potentially connecting top-down proteomics and HDX. The ICR group should avoid applications that can be performed routinely with commercial instruments of other types.

*Complex Mixtures*

- Advances in state-of-the-art ultrahigh resolution/high mass accuracy mass spectrometry will significantly improve the depth and breadth of complex mixture analysis. The data can be used for molecular characterization of oils, for understanding the biology of oil degradation in the environment and ultimately for development of a database of oils for source identification or for comparison of differences in chemistry. Ryan Rodgers described a future project where the toxic components of weathered fuels could be identified using state-of-the-art ICR MS. This project is exciting due to the intersection among the energy, environment and biology science drivers. Higher fields will increase the upper mass range of components that can be confidently characterized and the resolution of increasingly small mass differences.
- The ICR applications for the energy and environment science drivers are predominantly academic users. In addition, the Future Fuels Institute was established in 2012 and is a major user of the ICR facility.
- Petro Org is a commercial program developed at the NHMFL for petroleomic data analysis. Although the NHMFL retains the intellectual property, freeware versions are available for users to view their data. Adding quantification to the data viewing software is an important feature for future development. The ability to analyze data off-site was a concern in the user survey. Improvements in the distribution of data analysis software and data management are closely tied with the hiring of the informatics director.
- The ICR UAC noted the exceptionally strong performance of the petroleomics/complex mixture analysis program.

**(iv) Personnel**

- Personnel are a critical issue for the ICR program going forward.
- Hiring of an ICR Director and Biological applications Director is critical in the next 12-18 months.
- Over the long-term, there is a need for hires in informatics and instrumentation.
- Adequate technical staff is in place to meet the users' needs.
- A long-term strategy would be to coordinate a search with the Biology Department or Medical School for senior leadership in biological applications that take advantage of ICR.
- The environmental work is clearly going to be a growth area for the ICR User Program. With the amount of work already being performed in petroleomics, the ICR User Facility should consider adding an environmental chemist to their staff to relieve the pressure on this subgroup. This staff member should have training in environmental chemistry (or a related earth science field), help set strategic priorities and liaise with user.

**(iv) Priority list of Recommendations and Executive Summary**

- We recommend the hiring of two positions - an ICR Director and a Biological applications Director.
- The UAC and management need to propose a long-term plan to coordinate a search with host institutions for a senior faculty position in biological applications of ICR (e.g., through a medical school or Biology Department)
- The hiring of an environmental chemist to assist the petroleomics group would be worth exploring.
- A separate summer school for ICR techniques should be implemented
- ICR should solicit members of the UAC and select members of the user community for new proposals to UCGP.

**(v) Comments Related to the NSF Site Visit**

- The UAC endorses the ICR program's development of highly innovative applications in the areas of energy and the environment. The UAC felt that the energy/environment applications are a unique strength of the ICR program and represent an area of high growth.
- ICR expertise should be included on the NSF site visit team at annual and recompetition evaluation of the NHMFL.