EUROPEAN SPALLATION SOURCE

<<STAP Report Reflectometry June 2016>>

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Minutes

Meeting Date
14-15 June 2016

Location
Institute Laue Langevin, Grenoble

Chairman
Robert Cubitt

Secretary

Attendees
STAP: John Ankner, Karen Edler, Richard Campbell, Peter Mueller-Buschbaum, Mike Fitzsimmons.

FREIA instrument team: Hanna Wacklin ESS Clara Lopez ESS, Jim Nightingale ISIS, William Halcrow ISIS

ESTIA instrument team (PSI): Artur Glavic, Jochen Stahn, Sven Schütz.

ESS:
Prof. Andreas Schreyer,
Ken Andersen, Gabor Laszlo

Absentees

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STAP Report Template

1. Scientific case for neutron reflectometry at ESS

The STAP regards the scientific case outlined in a previous document as still relevant and it remains a valid baseline for the following recommendations.

2. Experimental requirements for reflectometers at ESS

The main technical requirements to fulfil the scientific case include measurements of air/liquid, liquid/liquid and solid/liquid interfaces and of liquids under shear, magnetic samples, magnetic reference layers, sub-minute kinetic measurements, efficient measurements on the minute time scale, reflection up and down over a broad dynamic range for horizontal samples, in plane structures – length scales from 1 nm to 10 um using off-specular scattering and grazing incidence small-angle neutron scattering (GI-SANS) – flexible resolution options and a world leading suite of sample environments as well as superb support laboratories.

Very often in instrument design projects (e.g. for FIGARO) the necessary sample environment is not foreseen in the initial budget. Because sample environment is highly specialized for reflectometry, e.g., to enable scattering at grazing angles, to minimize background, to accurately measure surface temperatures of films, to inhibit accumulation of condensates on surfaces, to enable operando measurements as functions of E, B, P and T, etc., the STAP considers sample environment to be an integral part of a world class reflectometer. For example, as important as the detector and as such sample environment should be rated as a high priority in the minimum acceptable scope for each instrument. The STAP is pleased that the instrument teams share this opinion but points out that soft matter sample environment is missing from the cost exercise, and the plan to achieve extremes of E, B, and P is not nearly ambitious enough as presented by the ESTIA team.

On this topic, the STAP pointed out that finally the ILL is correcting its long running mistake: its sample environment group was effectively a cryostat group with little or no support for the complex pieces of specialized equipment required for soft matter. After constant feedback from the users over many years, finally the culture at the ILL is now changing, and the central support for specialized equipment for soft matter will improve. In order to avoid a potential backward step, the STAP recommends the nature of support for specialized sample environments to be reconsidered by the ESS management. Their complexity should not be underestimated.

3. ESTIA focusing reflectometer

Which parts of the scientific case should be covered by this instrument, and how is it complementary to FREIA?

ESTIA will be able to measure a large fraction of the scientific case for reflectometry with the exception of measurements involving horizontal surfaces. ESTIA differs from FREIA in that ESTIA is optimized to perform reflectometry from samples of 1cm or less, and includes a polarized beam capability to study magnetic structures. The ability to measure small samples is important for reflectometry of quantum condensed matter because competition between different order parameters (which if exploited may have dramatic technological implications, e.g., enabling neuromorphic computing) nearly always requires interfaces with unit cell perfection, and samples with such perfect interfaces can not be readily grown with lateral dimensions much more than a few mm. In addition, some complex sample environments tend to require smaller samples, e.g., a pressure cell accommodating a 25 mm^2 reflectometry sample requires in excess of 40 tons to create 2 GPa. Subject areas include battery cells, electrochemistry, high pressure studies, lubricants, in plane structures by scanning large samples, laser flash induced kinetics, electric fields, fuel cells and microfluidics. The high flux mode will also provide outstanding time resolution for kinetic studies in all of these areas. Furthermore, a strong overlap between the ESTIA and FREIA instruments will fall in soft matter science in studies of proteins, polymers, surfactants, etc, at the solid/liquid interface. A particular strength of ESTIA will be the use of specifically designed miniaturized solid/liquid cells for the study of precious biological samples.

To what extent does the proposed instrument layout and technical solutions cover this scientific case?

The proposed instrument, if constructed fully, covers all of the scientific areas above. It will not be possible to use ESTIA for free liquid surfaces. A concern of the STAP is if cost cutting results in no provision of GI-SANS on either instrument on day 1. The technical implementation on ESTIA is more straightforward (lack of additional upstream slits) while the scientific case for FREIA is stronger (additional measurements on free liquid surfaces). The implementation on the two instruments also covers different length scales (ESTIA emphasizes lower Q than FREIA) so ideally both instruments should have GI-SANS capability.

Are the proposed technical solutions sound?

The guide alignment precision is a critical parameter in the instrument construction and maintainability. However, the ESTIA team is well aware of this constraint and have invested considerable effort in designing an appropriate system and completing a risk assessment. It is essential that the ESS provides a floor that is as stable as possible. The ESTIA team has also taken seriously the issue of effect of gravity in enlarging the beam at the sample with simulations already carried out, and are confident that the effect of gravity on the size of the focal spot is minimal.

Some concern was expressed about the high Q resolution option. Within the range of the natural resolution there would still be contamination of unwanted wavelengths. Simulation of this effect should be carried out to ensure that this does not introduce significant systematic errors into the reflectivity data when measuring close to high reflectivity gradients.

The quality of the detectors is still undefined in terms of their dynamic range, internal scattering, efficiency and resolution.

Is the project plan mature and realistic?

Yes. The STAP congratulate the ESTIA team members for their efforts.

What sample environments and support facilities are needed by ESTIA during hot commissioning and the first 5 years of user operations?

ESTIA requires routinely a magnetic field perpendicular to the sample's surface normal with variable temperature cryostats. The SNS experience is that the availability of a 5 T field is already critically needed (at least three experiments in the past year require greater than 4 T to saturate and with the community's keen interest to study systems, e.g., iridate composites, with strong L-S coupling and large U, more studies are foreseen immediately). The Asterix at Lujan experience using a "pool" 11 T cryomagnet for reflectometry—one that did not have special low background cold windows—was that the background was increased by a factor of at least 10 in a range of Q restricted to <0.2$Å^{-1}$. The HFIR at ORNL experience with "pool" cryomagnets for studies of thin films with SANS was that such studies simply can not be done. Yet, at NIST with a cryomagnet designed for SANS experiments, e.g., one with single crystal cold windows, SANS studies of thin films have been very productive. To accommodate the hard-matter reflectometry science we see demand now and expect only to increase in 10 years time, for a high (>5 T) magnetic field and cryogenic solution customized for reflectometry (and/or SANS) will be critical. Generally speaking the ESTIA instrument's proposal for sample environment lacks other critical capabilities. For example, a vector field cryomagnet is required (e.g. one with 2 T out of plane capability—the out of plane capability is required to realize novel states of quantum condensed matter, skyrmions…), a 2 GPa pressure cell compatible with cryogenic temperatures, means to apply electric fields to samples, to measure transport in situ, e.g., anomalous Hall effect, microwave generator to excite spin waves, magneto-Kerr-effect spectroscopy, etc. The latter examples are ones that enable multi-modal experiments, which are becoming the norm in the synchrotron community and emerging in the neutron community. In 10 years time multi-modal experiments may likely be the norm for neutron reflectometry.

The ESTIA team have not mentioned the soft matter sample environment in their costing with the exception of sharing equipment with FREIA (see section 5.2.5 of the *Concept of Operations* document, 27/05/2016). This raises two issues. First, if there is a realistic possibility that ESTIA will come online before FREIA then the STAP encourages the ESTIA team to communicate well with the FREIA team in the specification of what is required and even take the lead in the development. This issue not only needs to be included in the costing but investment will be required in gaining the appropriate expertise to design cutting edge small samples cells ready for day 1 that will probably be used only on ESTIA. Other ancillary items will be required by the first users of ESTIA such as an HPLC pump, HPLC value, syringe pump, water baths etc. If these items are pooled equipment, the ESS should consider that the sample environment group can provide it. Second, it is often the case that on FIGARO and D17 there are solid/liquid interface experiments in parallel. Therefore sufficient equipment for each instrument and spares are required. It will not be realistic to rely only on using FREIA's equipment. Users will require a small chemistry lab for sample preparation.

At the SNS and NIST we see examples of neutron scattering experiments being performed in conjunction with computational modelling in real time. Examples include applications of density functional theory or molecular dynamic simulations. The ESTIA proposal lacks a plan on how to integrate computational modelling into data visualization and analysis. More generally the ESS is encouraged to develop a strategic plan to realize integration of high performance computing into neutron scattering experiments.

What are the scientific priorities for realising the instrument's scientific scope?

The crucial strong point of ESTIA, which will open up genuine new areas of science, is the ability to produce a tiny beam of neutrons, on the scale of 1-10 mm and can measure reflectivity extending over a modest range of Q on a time scale of seconds or less. The implementation of these aspects will allow ESTIA to cover the scientific case as specified above.

What experimental capabilities should be available on day 1 and what could be brought in through upgrades?

The scientific priorities for realizing the instrument's scientific scope are as follows in order of priority:

1. High-intensity specular and off-specular reflectivity for vertical interfaces including sub minute kinetics and <cm size (general instrument framework, e.g. guide system (with all the vertical mirrors), metrology and detector)
2. Studies of magnetic samples (polarization option with analyser)
3. Sample environment (soft matter and hard matter)
4. Wide bandwidth to aid kinetics (chopper)
5. GI-SANS (re-focussing mirror)
6. Very small footprint measurements <<mm i.e. microfluidics (Ultra-precise focusing)

What experimental capabilities should be available on day 1 and what could be brought in through upgrades?

The STAP considers the minimum acceptable scope for day 1 in order for the ESS to go ahead with the ESTIA instrument is the first 3 items on the list immediately above. This will allow conventional specular reflectivity experiments to be carried out using a range of sample environments, but it is highly likely that measurements of very small samples (< cm) will be compromised if the metrology system, virtual source aperture and ultra precise focusing are not in place at the same time, ESTIA's selling point from the outset. The STAP strongly encourages the provision of items 4–6 as well. The absolute necessity for GI-SANS on ESTIA is coupled with the decision about its provision on FREIA although there is a need for both free liquid and low Q provision.

We will be asked to present the following at the scope setting meeting:

<u>A) Scope within cost category:</u> Which parts of the scientific case are met and to what extent is the instrument configuration realised within the cost category of 9M€ (incl. 10% contingency)? What upgrade paths are possible to achieve configuration B or C?

The instrument team were forced to sacrifice more items than the acceptable minimum scope of the STAP listed above to reach the target of 8,100,000 euros. The most serious is the loss of the majority of the vertical mirrors and the double beam leading to x30 loss of neutron transport and an instrument close to the performance of D17 the ILL. In addition this cheap version lost most of the sample environment, chopper, virtual source, metrology system, magnet, cryostat, flight tube, analysers & polarizers. The STAP considers this outcome to be unacceptable and such a *severely compromised instrument* is not worthy of the ESS. The STAP believes the instrument was not placed in the correct cost category.

<u>B) Minimum acceptable scope (if not A):</u> What is the minimum acceptable scope that covers the essential day 1 science and allows for later upgrades to the full instrument scope?

Please see the comment above about what the STAP considers must be present on day 1 i,e the first three items.

<u>C) Full scope of instrument (if not A or B):</u> What is this and how can it be achieved by upgrading from the day 1 scope of the instrument (A or B)?

The full scope of the instrument, presented by the ESTIA instrument team comprised of all the items listed. The STAP feels that the staging of any items not present on day 1 must be anticipated in the initial design so as not to present high barriers or incur unnecessary wastage.

4. FREIA horizontal reflectometer

Which parts of the scientific case should be covered by this instrument, and how is it complementary to ESTIA?

The FREIA team expect the instrument to cover the following areas: soft condensed matter (surfactants, polymer physics, electrochemistry, templated nanostructures, air/liquid, liquid/liquid and solid/liquid interfaces), life sciences (pharmacy, protein behaviour and membrane physics, biointerfaces), energy research (display screens and OLED technologies, hydrogen storage and lithium intercalation), environmental sciences (ocean surfaces, biointerface/liquid/solid surface response to airborne & aqueous pollutants, cloud droplet model systems) and engineering materials (surface chemistry of metals). We agree that the technical requirements proposed for the full instrument will allow all of these scientific areas to be researched.

Much of the scientific case for FREIA involves studies of samples that generally can be made much larger than those involving quantum condensed matter. As such there is much less of a need to produce highly focused beams that illuminate very small samples. The majority of samples studied with reflectometry can be made larger than 1 cm^2, so our expectation is most of the soft matter program, including kinetic studies, will be best accommodated on FREIA.

To what extent do the proposed instrument layout and technical solutions cover this scientific case?

The proposed instrument, if constructed fully, covers all of the scientific areas above. It will not be possible to use FREIA with high magnetic fields (due to its proximity to a spin echo instrument). Further, FREIA will not excel at the reflection down (upward directed incoming beam) option for horizontal samples over a broad dynamic range, its performance will be compromised for small samples with a lower limit of ~1 cm^2 and the GI-SANS option will have a limited dynamic range. We have no concerns about the first two issues because ESTIA will excel with high magnetic fields and with small samples. However, we do have significant concerns that an optimized instrument for GI-SANS and reflection down is not included in the initial instrument suite at the ESS. Each issue is addressed in turn.

The primary concern of the STAP is that there will not be an optimized GI-SANS capability compatible with specular reflectivity measurements – in particular of free liquid surfaces. We anticipate a strong demand for this combination making the GI-SANS option on a neutron reflectometer more suitable for FREIA than ESTIA. The STAP also discussed the alternative of deflecting the beam down on a SANS machine, which at present is foreseen for reflection angles that are too small to perform neutron reflectivity from free standing liquid surfaces as well, and the associated sample environments necessary for such measurements are unforeseen (the absence of sample environment for soft matter was also noted by us for the ESTIA proposal). The STAP recommends strongly that the GI-SANS option is installed on FREIA.

A second concern is the lack of an optimized reflection down option to achieve the full dynamic range foreseen on FREIA. Currently on FIGARO approximately half of the experiments use this option: free liquid/liquid interfaces, rheometry and the majority of solid/liquid interface experiments. The former two categories require transmission through the lower phase followed by reflection down at a horizontal interface. A broad dynamic range is required for fluorocarbon oils at liquid/liquid interfaces and through solid substrates for rheometry. The latter category uses a beam inclined only slightly upwards and then the sample can be tilted to adjust theta. Note that the reason users prefer to reflect down from a solid/liquid interface is that any air bubbles that are trapped or form in the liquid cell can float away from the probed interface. The possibility to reflect the beam downwards from day 1 is strongly encouraged, as it may be the case that the majority of users wish to use this option.

The STAP therefore recommends strongly that the GI-SANS and reflection down options are available on FREIA and in addition that the ESS continues to invest in the design of a dedicated instrument to cover both missing parts of the technical requirements: reflection down over a broad dynamic range and optimized GI-SANS.

Are the proposed technical solutions sound?

In the main, yes, the technical solutions proposed are sound. Some issues remain:

The technical requirement of the fast shutter system has been defined as using three consecutive pulses at different incident angles on the sample to cover the full dynamic range in a fraction of a second. A key unknown is its performance and more work is required on the design. Simulations of 70 ms data should be carried out to justify this technical requirement because if more than 3 pulses are required (e.g. at the highest incident angle) the engineering specifications of the system could be relaxed.

The STAP recommends that the entire slit system should be easily removable so the full divergence of the beam can be used in the same manner as the high flux mode of ESTIA. Calculations for the shielding implications of this mode to enable its feasibility should be carried out.

The quality of the detectors is still undefined in terms of their dynamic range, internal scattering, efficiency and resolution.

Is the project plan mature and realistic?

The STAP appreciates the efforts that the FREIA team have made to adapt the instrument design to the new moderator design, and the associated benefits. Although scientifically mature, the FREIA instrument concept is technically less mature than that of ESTIA. The anticipated budget and timeline for design, installation and commissioning, for example, still has to be carried out.

What sample environments and support facilities are needed by FREIA during hot commissioning and the first 5 years of user operations?

The FREIA team presented the essential (air/liquid adsorption troughs, Langmuir trough, set of solid/liquid flow cells, liquid/liquid cell, HPLC pump + syringe pump and 2 water baths) and optional (humidity chamber, electromagnet, vacuum chamber and furnace) sample environments that are required for day 1. The STAP agreed with this list but suggests that also a set of bulk liquid/liquid sample cells will be standard equipment by then and so should be considered essential.

In the commissioning of FIGARO, it took almost one year of technical design and manufacturing to have the air/liquid adsorption troughs ready for day 1, and even they were based on existing designs from ISIS. The next phase of sample environment development on FIGARO will begin in September 2016 when there will be a 200,000-euro investment to design a suite of optimized adsorption troughs with in situ surface tension monitoring and a new suite of various Langmuir troughs and containers. The ESS Science Director suggested that this could be a project in which the ESS could collaborate with the ILL, and the instrument responsible of FIGARO was openly receptive to this idea. This could be a way in which FREIA can make significant progress towards the provision of optimized sample environment ready for day 1.

With regards to the solid/liquid soft matter sample environment, the STAP encourages the FREIA and ESTIA teams to communicate with each other in coordination of the design and construction of sample cells and purchase the necessary ancillary equipment (e.g. HPLC pumps, HPLC valves, syringe pumps, water baths etc.) between them ready for the first neutron reflectivity user experiments at the ESS. The issue of communication was also raised in terms of the FREIA and SANS teams concerning the acquisition of a rheometer and cryomagnet. These pieces of equipment have a similar price, including all the necessary accessories, and would be used on both the SANS instruments as well as reflectometers. It should be clear where the funding would come from. Finally, if magnetic reference technology is to be exploited, a method of saturating these layers will need to be provided, and a means to polarize the incident neutron beam (and analyse its polarization after reflection).

What are the scientific priorities for realising the instrument's scientific scope?

The scientific priorities for realizing the instrument's scientific scope are as follows in order of priority:

1. High-intensity specular and off-specular reflectivity for studies at the air/liquid and solid/liquid interface including sub minute kinetics (general instrument framework, e.g. guide system and detector)
2. Sample environment
3. High, medium and low resolution options for specular and off-specular reflectivity due to the wide range of length scales to be studied, particularly in soft matter (WFM choppers)
4. Lateral structures over a range of length scales (GI-SANS)
5. Free liquid/liquid interfaces and rheometry (reflection down configuration)
6. kinetic measurements over a broad dynamic range requiring the fast shutter system.
7. Magnetic reference layers (polarized neutron reflectivity)

What experimental capabilities should be available on day 1 and what could be brought in through upgrades?

The STAP considers the minimum acceptable scope for day 1 in order for the ESS to go ahead with the FREIA instrument is the first 3 items on the list immediately above. This will allow conventional specular reflectivity experiments to be carried out for the majority of the science case. Nevertheless, the STAP strongly encourages the provision of items 4–7 also from day 1 as this is a combination of new capabilities and existing science that will otherwise be missing. Specifically, if GI-SANS were to be cut from day 1, the implementation path should not involve a significant re-building of the instrument, i.e. the detector tank should have a sufficient size or be modular in design so as to accommodate a large detector, and the walls of the sample area should be installed initially allowing for the necessary sample-to-detector distance. The STAP is concerned that even moving a wall can cause great administrative issues regarding nuclear safety authorities. Also, space for the GI-SANS slits in the bunker should be foreseen. The implementation of the reflection down option should be foreseen and straightforward to install.

The STAP, however, would like to emphasize that a GI-SANS option on FREIA should not in any way reduce the efforts that the ESS must make in the design and implementation of a dedicated instrument for optimized reflection down and GI-SANS measurements both in terms of a broad dynamic range and maximized brilliance transfer.

We will be asked to present the following at the scope setting meeting:

<u>*A) Scope within cost category:*</u> *Which parts of the scientific case are met and to what extent is the instrument configuration realised within the cost category of 9M€ (incl. 10% contingency)? What upgrade paths are possible to achieve configuration B or C?*

Costing estimates for FREIA are not yet ready, so the STAP cannot comment definitively on this issue. Nevertheless, it was perfectly clear to the STAP that an instrument as powerful and flexible as FREIA – *covering the vast majority of the scientific case for reflectometry* – has been placed in the wrong cost category. There are significant genuine additional costs for FREIA over ESTIA, including the choppers and shielding, the combined difference of which is estimated to be in excess of 4,000,000 euros. As such, the predicted budget of FREIA is currently 15,850,000 euros to deliver the full scope set out by the STAP. This difference seems to have been missed by the ESS when allocating the instrument in its provisional price category of 9,000,000 euros. Therefore it seems absolutely unrealistic to perform the cost-cutting exercise during the STAP meeting with a target of 8,100,000 euros, especially given that FREIA and ESTIA are unsupported for sample environment in contrast to most instruments that will benefit from pooled cryostats and magnets. Taking these issues into account, the STAP considers it unrealistic prospect that the ESS are asking the FREIA team and the STAP to assess what is possible at half price. The STAP reserves judgement on this issue until the appropriate costing has been carried out, but it looks highly likely at this stage that the performance of FREIA will not be close to meeting the minimum acceptable scientific scope defined above with a budget of 8,100,000 euros.

<u>*B) Minimum acceptable scope (if not A):*</u> *What is the minimum acceptable scope that cover the essential day 1 science and allows for later upgrades to the full instrument scope?*

Please see the comment above about what the STAP considers must be present on day 1.

<u>*C) Full scope of instrument (if not A or B):*</u> *What is this and how can it be achieved by upgrading from the day 1 scope of the instrument (A or B)?*

The full scope of the instrument has been presented by the FREIA instrument team and includes all of the items on the list above. The STAP feels that the staging of any items not present on day 1 must be anticipated in the initial design so as not to present high barriers or incur unnecessary wastage.

5. Complementarity and overlap

Complementarity and overlap of the two instruments: which experiments, including sample environments, resolution options etc. should be available on both instruments and why?

The complementarity and overlap of the two instruments have been thoroughly discussed above. The instruments are highly complementary and between them they will deliver the majority of the scientific case for reflectometry defined during the first SAP meeting. The major shortcoming has also been discussed above: that is, even if ESTIA and FREIA are built in full, neither are optimized for reflection down and GI-SANS over the full dynamic range with maximized brilliance. The consequences for the scientific output of the ESS are far reaching and are discussed in more detail below.

6. Future instruments

What (additional) experimental capabilities should be covered by future surface instruments at ESS and what are the scientific priorities?

Should these capabilities be incorporated into ESTIA/FREIA on Day 1 or brought in as upgrades?

As a result of the shortcoming mentioned above, the STAP for reflectometry consider it to be essential that the ESS management make it a *high priority* to go ahead urgently with the designs and scope setting for a surface scattering instrument with optimized performance for GI-SANS and reflection down over a broad dynamic range with maximized brilliance. It is important that both options are available on FREIA on day 1. GI-SANS at the ESS will undoubtedly open up a great deal of new science in terms of lateral interactions, and the provision of a lateral density profile to complement the information normal to the interface. This will be especially important for biological systems including membrane proteins (especially complex formation or increasingly concentrated, multicomponent systems) and lipid in-plane inhomogeneity but will also impact on synthetic soft matter experiments such as polyelectrolyte multilayers, mixed amphiphile adsorption at oil or air-water interfaces (relevant to, for instance, personal-care and homecare products), experiments on reacting systems (e.g enzyme interactions with lipid layers, pollutant degradation of lung surfactants, formation & degradation of adsorbed organic monolayers on seawater) and nanoparticle interactions. The increased need to study complex mixtures and concentrated systems is already evident in reflectometry experiments, and the requirement to obtain lateral information to determine in-plane phase separation, clustering and self-assembly is essential to allow realistic data modelling of even the specular reflectivity from such systems. Furthermore, as the majority of experiments currently performed on FIGARO use the reflection down option, the STAP anticipates that this could also be the case on FREIA. It will also allow the next level of science on liquid/liquid interface and rheometry, which are becoming popular these days and will simply be expected by the time FREIA comes online. The provision of these options on FREIA from day 1 is very important. Nevertheless their implementation must in no way diminish the need for a dedicated instrument to provide these two capabilities in an optimized way as soon as possible afterwards.

The STAP recognizes opportunities to leverage development of sample environment for hard and soft matter programs between the reflectometers and SANS machines, and with other neutron scattering facilities.

The STAP recommends ESS be proactive in development of sample environment. The STAP also recognizes growing opportunities to integrate high performance computing into real-time analysis of neutron scattering data. The STAP recommends ESS be proactive in these areas by organizing collaboration meetings along the lines as done for accelerators (e.g., the International Collaboration on Advanced Neutron Source, ICANS).