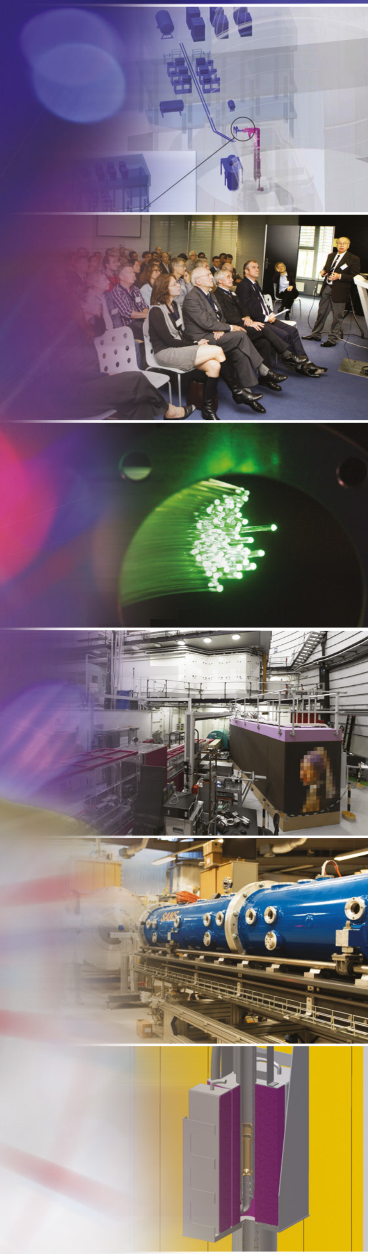
2015

OYSTER

Annual Report

Celebrating PEARL



TU Delft Delft University of Technology

REACTOR INSTITUTE DELFT

2015

OYSTER

Annual Report

Celebrating PEARL

Foreword

It is with pleasure that I present this OYSTER annual report to you.

The new beam instrument PEARL has been taken into operation during OYSTER's fourth year and has generated a great deal of interest, as also showed by the large attendance at the introduction meeting at the RID institute. PEARL's new smart design means it is comparable with the best neutron powder diffractometers in the world, including those with neutron sources ten times as powerful. This new instrument is improving energy material research. Researchers are able to conduct scientific research into energy storage materials for, for instance, wind and solar energy, and will be able to better investigate clever cooling materials, such as for magnetic cooling.

The POSH-PALS instrument is excellently suited for revealing the behaviour of defects, as e.g. in thin-film solar cells. A new test chamber has been installed and measurements will be performed on real samples by the end of this year.

Other achievements for 2015 include the design of a flexible irradiation facility. The acquired additional funding means we will be able to build and install a prototype this year.

The SANS instrument reaches completion ahead of the installation of the cold source. SANS is a technique typically used to investigate proteins, micelles, polymers, porous media and precipitates.

The basic design of the cold neutron source (CNS) - to be built in our reactor – was worked out in further detail following the selection of the KHC consortium. The cold neutron source will allow us to increase the intensity of low-energy neutrons in order to improve the sensitivity of the beam instruments. The design for the CNS Utility building was finalised and the associated cooling equipment requirements were determined as the basis for the next phase in the implementation of the OYSTER project. RID supported the consortium in the search for an experienced, well-known local supplier to realise the CNS Utility building and associated equipment.

This annual report gives you an insight into these and the other great things that are happening in various areas within the OYSTER program and gives me confidence that 2016 will again be a year full of highlights.

April 2016

Prof. dr. Bert Wolterbeek
Director of the Reactor Institute
Delft Head of the Department
on Radiation, Science &
Technology

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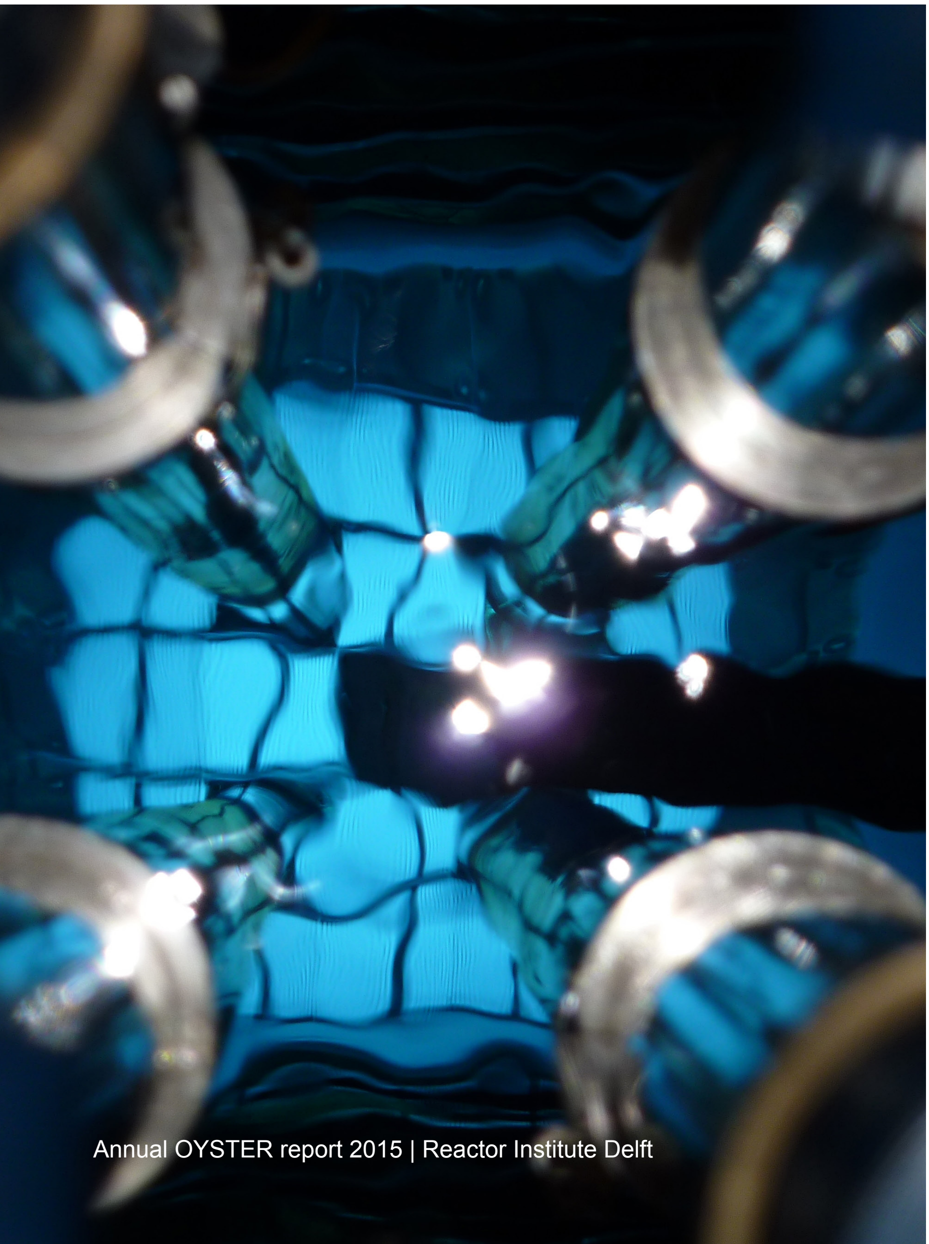
OYSTER in short

A general introduction (pages 4-7) to the OYSTER programme and RID.



The OYSTER programme (Optimized Yield - for Science, Technology and Education - of Radiation), co-funded by the Dutch government, TU Delft and a number of commercial parties, is to expand the potential of the research reactor by improvements and expansions of the RID infrastructure (reactor, instruments, facilities).

This expansion will enable current and future educational, scientific and societal questions to be better addressed. RID also contributes in-kind, through OYSTER, to the ESS (European Spallation Source in Lund, Sweden), by means of the development of neutron instruments.

The Reactor Institute Delft

The TU Delft Reactor Institute Delft (RID) is a nuclear knowledge centre. It operates the reactor, the irradiation facilities and laboratories, as well as neutron- and positron instruments. In conjunction with the Department of Radiation Science & Technology (RST) of the Faculty of Applied Sciences, RID accommodates resident and visiting scientists and other users from a variety of (scientific) disciplines. RID educates students, professionals and scientists, and serves as an independent source of information for society on radiation- and nuclear-related issues.

The technique

The installation of a Cold Neutron Source (CNS), which will cool neutrons from room temperature to -250 ºC and will therefore increase the intensity of low-energy neutrons by more than an order of magnitude. This will improve the sensitivity of existing top-class instruments.

The design and construction of new research instruments.

The (re-) design and construction of (new) irradiation facilities, which permit the (development of) production of radioisotopes with unprecedented purity and which will increase the sensitivity and opportunities for research with isotopically enriched stable isotopes.

The design and installation of a miniature hot-cell/decanning facility for submerged access of irradiated samples from the irradiation facilities. The hot cell serves mainly as a decanning facility of canisters containing samples which have been irradiated for the research programme by innovative production methods of (medical) radioisotopes. It is also employed for sub-sampling of these samples to study radiation damage effects.

The design of irradiation facilities positioned in the tangential beam tube of the reactor, to undertake research into alternative production methods for e.g. Molybdenum-99 (99 Mo).

Participation in large international and national collaborations

RID participates in the development of the European Spallation Source (ESS, www.EuropeanSpallationSource.se) in Lund, Sweden, which is an international collaborative facility for materials research using neutron scattering techniques. The Dutch contribution to the pre-construction phase of the ESS is partly financed through OYSTER. For this purpose RID works on the development of novel instrumental concepts for the ESS and in close collaboration with the ESS scientists.

RID participates in the R&D of Holland Particle Therapy Centre (Holland PTC, www.HollandPTC.nl) dedicated to innovative radiation treatment of cancer, using proton beams, as well as in a collaborative R&D program at TU Delft, the Leiden University Medical Centre (LUMC) and the Erasmus University Medical Centre Rotterdam (Erasmus MC). HollandPTC is under construction and will be located on the RID premises.

OYSTER research also strengthens the role of RID in supplying innovative ideas towards the envisioned PALLAS reactor (www.pallasreactor.com). This is to become one of the world's leading production sites for medical isotopes.

The OYSTER-initiated new irradiation facilities enhances RID's position in DIVA (Dutch Isotope Valley). This is an R&D collaboration set up between URENCO, RID and NRG/PALLAS, aimed at optimizing efforts to develop, engineer and produce the best possible medical isotopes for clinical use in both (radio)diagnostic and (radio) therapeutic hospital protocols.

Finally, OYSTER underlines the role of TU Delft's RID as an International Atomic Energy Agency (IAEA) Collaborating Centre by demonstrating the many innovative scientific opportunities in the utilization of a medium-sized university research reactor.

End of general introduction.

Start year report on page 10 >>

Our five main goals

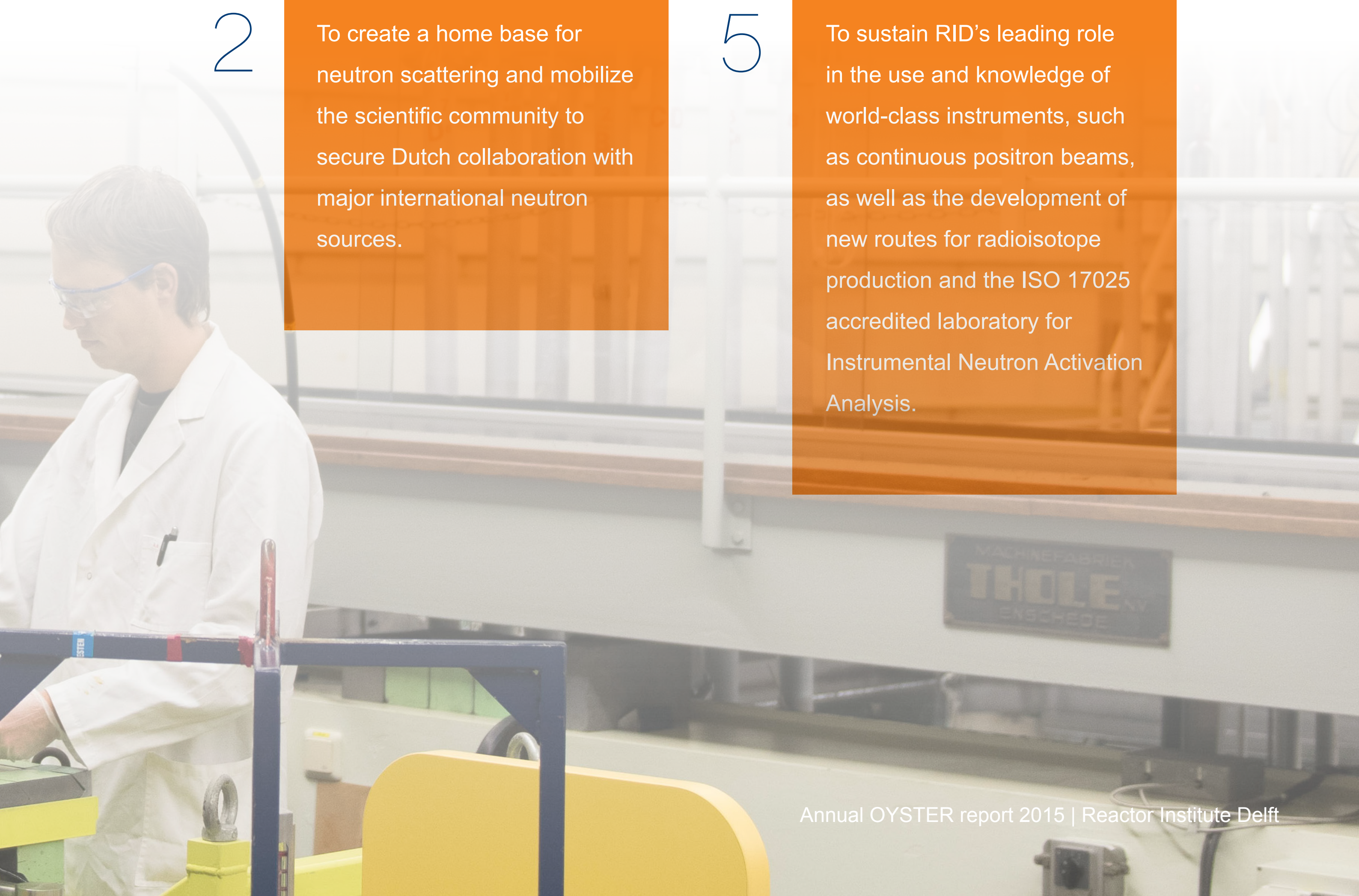
1 To further develop RID as a coordinating centre for the application of neutron, positron and radiochemistry science and techniques, as well as radiation detection and reactor technology, thereby supporting and uniting the Dutch scientific community.

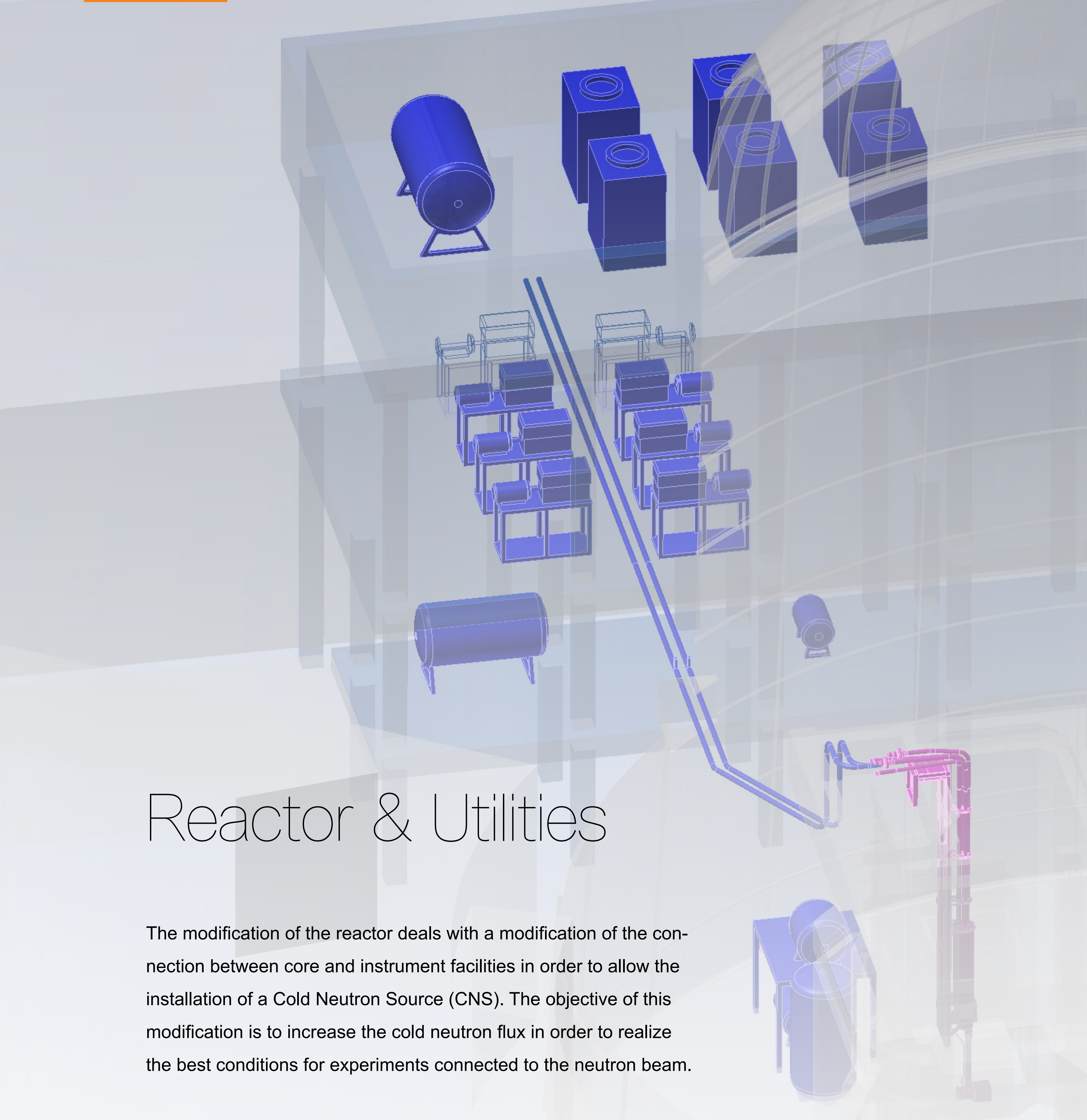
2 To create a home base for neutron scattering and mobilize the scientific community to secure Dutch collaboration with major international neutron sources.

3 To establish RID as a knowledge centre and training institute in Europe, and therefore a coordinating partner in European research networks.

4 To stimulate ground-breaking innovations in the field of neutron, positron, reactor and radiochemistry science.

5 To sustain RID's leading role in the use and knowledge of world-class instruments, such as continuous positron beams, as well as the development of new routes for radioisotope production and the ISO 17025 accredited laboratory for Instrumental Neutron Activation Analysis.





Reactor & Utilities

The modification of the reactor deals with a modification of the connection between core and instrument facilities in order to allow the installation of a Cold Neutron Source (CNS). The objective of this modification is to increase the cold neutron flux in order to realize the best conditions for experiments connected to the neutron beam.

Cold Neutron Source Utilities consist of the installation of all utility systems to the CNS which will be located outside the reactor building. This includes a new CNS Utility building with a cryogenic installation next to the Reactor hall and all associated equipment required to produce cooling capacity for cooling the Cold Neutron Source.

Delineation between scope of works

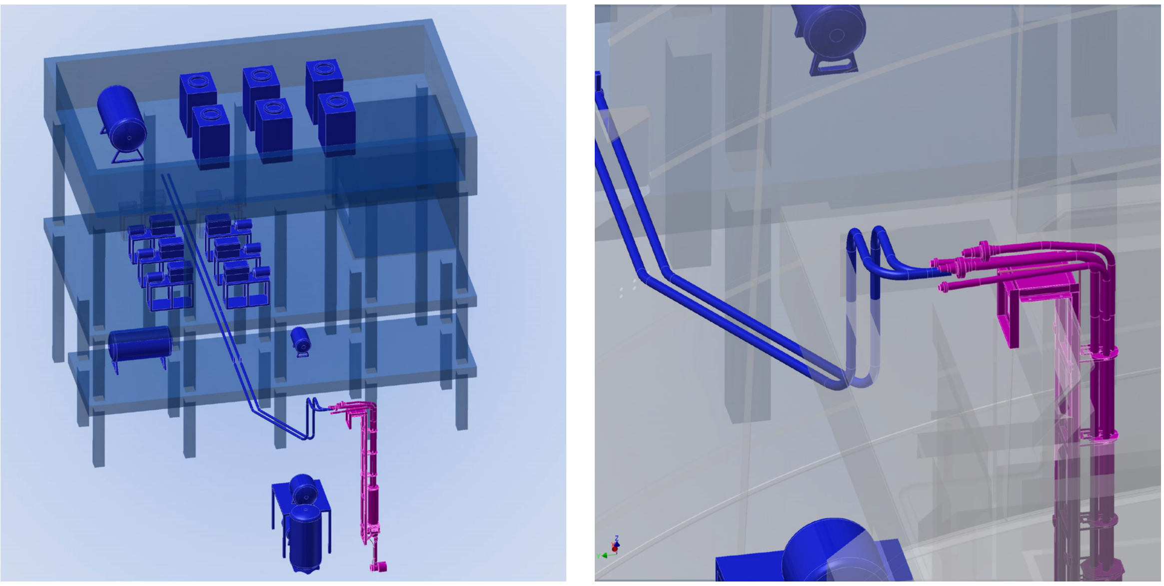
The delineation between the scope of works for the CNS Utilities and the In-pile component is an important aspect in the realization of the OYSTER project. The intention is that the realisation of the CNS building - including the cryogenic equipment - will be provided by local Dutch suppliers. The required cooling capacity must be according to the specifications described in the consortium KHC process design and will be guaranteed by the local contractor.

It is envisaged that the manufacture and installation of the CNS In-pile component (including the beam-tube modifications) will be undertaken by an international contractor with relevant construction experience with Cold Neutron Source equipment.

Basic and detailed engineering

Following the selection of the KAERI, Hyundai Engineering and Hyundai Engineering & Construction (KHC) consortium as the winning contractor for the OYSTER project, the basic design for the DSR related items was further worked out. An important issue here is the availability of the final version of the DSR - the new Dutch Safety Regulations - for modifying new and existing nuclear facilities. One important aspect of the DSR legislation is the requirement for the renewal of the primary/secondary cooling system for the reactor. Although other issues also have to be implemented, this item has a major impact on the price level for DSR-related costs.

The OYSTER contract agreement defines KHC's DSR-related cost proposal and the



Photos: The delineation between the scope of works for the CNS Utilities and In-pile component: the flange located on the platform railing of the reactor basin.

selection of (local) subcontractors for the work as criteria for a Go/No-Go decision by the Executive Board for further reactor modification. A start has also been made on the detailed engineering for the optional scope of work and the CNS Utility part. The CNS Utility building design was finalised and the associated cooling equipment requirements were fixed as a basis for the next phase in the OYSTER project: the procurement/construction phase.

Selection of Dutch and international subcontractor

RID supported the consortium in finding an experienced, well-known local supplier for realizing the CNS Utility building and associated equipment. In December 2015 a breakthrough was made with the selection of Royal Haskoning DHV (RHDHV) as subcontractor for CNS Utility works. This means RHDHV will become responsible for developing and realising the CNS Utility building, as well as providing the cooling equipment required for the requested cooling capacity. Furthermore, KHC selected an international, experienced company for the CNS In-pile component. The latter will become responsible for the installation of the Cold Neutron Source and the modifications to the beam-tube.

Responsibilities parties

A major effort was undertaken to delineate the responsibility issue, i.e. which party will be responsible for the functionality and performance of the CNS Utility works, the In-pile component and the overall guarantees as described in the contract. It was finally agreed that RHDHV will assume responsibility for the CNS Utility process performance conditions, and that the In-pile functioning will be the responsibility of the international company. The latter will undertake work on the Cold Neutron source and modifications to the beam tube. The contractual guarantee on flux- and neutrons performances will be provided by the KHC consortium. The division between the scope of work for the CNS Utility and the In-pile component is situated at the flange located on the platform railing of the reactor basin (inside the reactor building).

The external expert team

During the first half of 2015 the expert team was intensively involved in the review process for the design of the reactor modification. The specialists mainly focussed on the technical aspects related to the in-pile part and cold neutron source. The team made an important contribution to the final lay-out of this equipment.
The involvement of the expert team was limited in the second half of 2015 due to the start of the negotiation process. The discussions with the KHC consortium mainly focussed on procurement and legal aspects to arrive at a final agreement. The detailed engineering aspect for the cold neutron source facility will have to be produced once the negotiation process has been finalised. The role of the expert team will, therefore, increase significantly in 2016.

The external expert team

Stuart Ansell

During 2014 scientist at ISIS STFC (UK) specialized in Cold Neutrons equipment design for research reactors.

- Leading specialist for optimization processes neutronics

Robert Williams

Nuclear Engineer and Cold Neutron Source Team Leader at the National Institute of Standards and Technology (NIST) in Gaithersburg, Mary land, (USA)

- Cold source performance calculations, CNS operations and safety

Robert F. Mudde

Professor of Multiphase Flow, Department Chemical Engineering,
Delft University of Technology.

- Specialist in heat and mass transfer as well as hydrodynamics of multiphase flows.

Stephan Welzel

Chief coordinator reactor upgrade Helmholtz-Zentrum Berlin specialized in CNS process technology and operational aspects

- CNS process technology

Toni Scheuer

Nuclear Technology Consultant for TUV Rheinland Group.
Specialized in licensing issues, and material- and component qualification.

- Welding procedures, Materials, Codes & standards

Licensing

Since 2013, the required licensing procedures and associated review schedules for the OYSTER project approach have been under discussion with the regulatory body: the Authority for Nuclear Safety and Radiation Protection (ANVS). Part of the license application will be a Safety Report, a Safety Analysis Report and Environmental Impact Assessment.

Safety Report and Safety Analysis Report

A Safety Report will be part of the license application in order to demonstrate that the reactor including the OYSTER modifications can be operated safely. The supporting documentation for this report, e.g. detailed drawings, analysis reports etc., will be part of a separate document: the Safety Analysis Report. The discussions and drafting of these documents started in 2014, and part of the chapters has been sent to ANVS in 2015 for review. The reports will be finalised in 2017 and will be part of the license application for the new operating permit.

‘Environmental Impact Assessment’ (MER)

The preparation of the Environmental Impact Assessment (Milieu Effect Rapportage, MER) has progressed well. NRG was selected for the preparation of the MER in 2013 on the basis of the MER guidelines for the OYSTER project (Notitie Reikwijdte en Detailniveau). The first chapters of the MER were drafted and discussed with the ANVS in 2015. The MER will be finalised in 2016 and will be part of the license application for the new operating permit.

Dutch Safety Requirements (DSR)

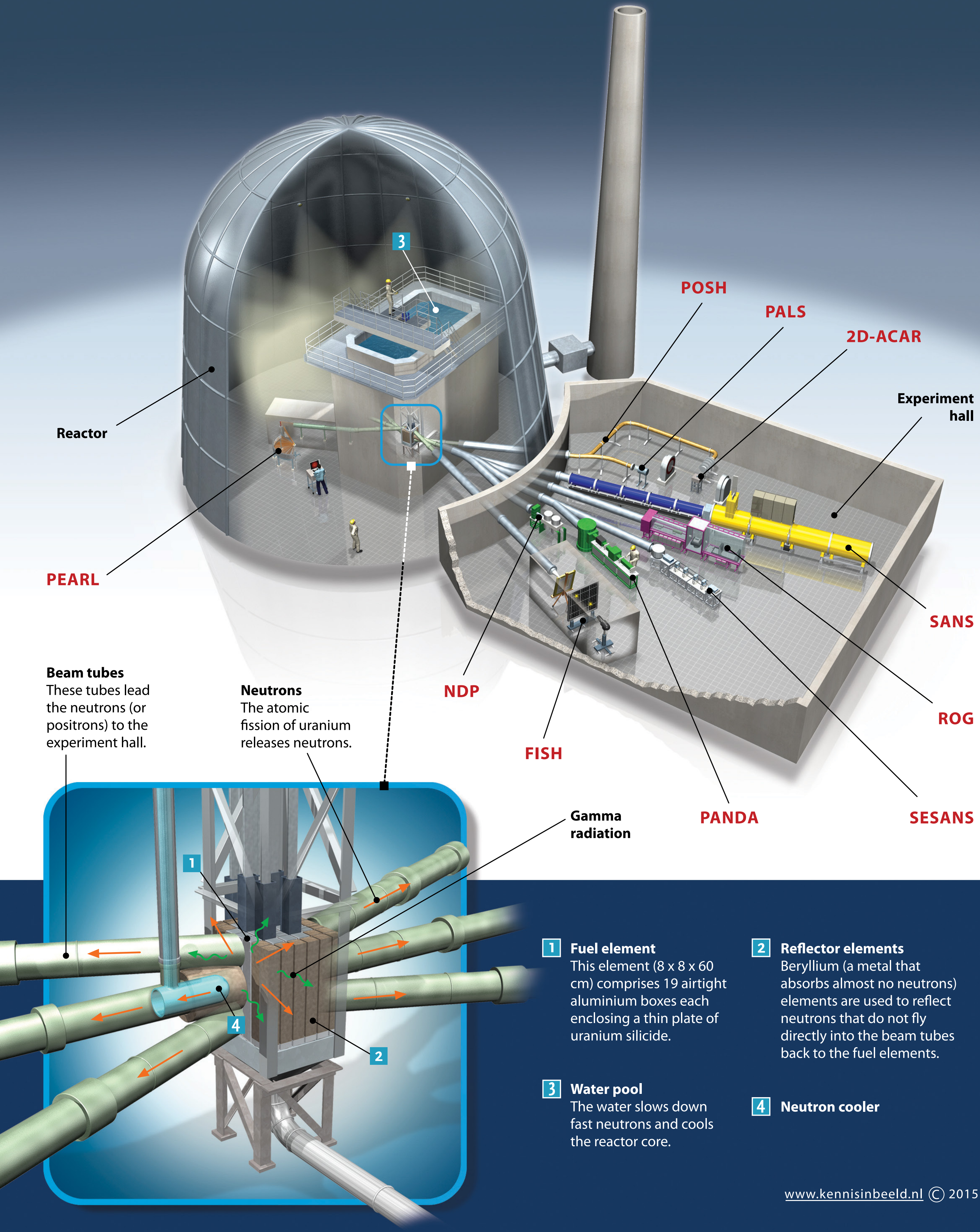
New and more stringent Dutch Safety Requirements (DSR) for new nuclear reactors were presented at a workshop in June 2013, and the Dutch nuclear community was invited to participate in the discussion with the Dutch regulator by providing advice and comments in terms of the further development of these new requirements. Finally, the formal publication of the DSR was in October 2015. Because the DSR legislation forms an important aspect in the contract conditions of the agreement between the consortium KHC and TU Delft, the project schedule for OYSTER will be delayed.

Photo: Aad van der Kooij.

OYSTER EXPANSION

Measurement instruments

around the research reactor



Beam tubes
These tubes lead the neutrons (or positrons) to the experiment hall.

Neutrons
The atomic fission of uranium releases neutrons.

1 **Fuel element**
This element (8 x 8 x 60 cm) comprises 19 airtight aluminium boxes each enclosing a thin plate of uranium silicide.

2 **Reflector elements**
Beryllium (a metal that absorbs almost no neutrons) elements are used to reflect neutrons that do not fly directly into the beam tubes back to the fuel elements.

3 **Water pool**
The water slows down fast neutrons and cools the reactor core.

4 **Neutron cooler**

Instruments

Short overview of instrument status in 2015 and an outlook into next year. See the next pages for a more detailed overview of each instrument.

PEARL – a neutron powder diffractometer (scientific opening workshop and first experiments)

- Building a growing user base in 2016.
- Development of required infrastructure for researchers: support, sample environment and easier access.

POSH-PALS -positron annihilation lifetime spectroscopy (under construction)

- New test chamber at the beginning of 2016.
- Tests and further optimisation of the timing system and the subsequent beam transport elements.
- First experiments on real samples expected by the end of 2016.

SANS – a new small angle diffractometer with a dedicated cold beam line (almost finished)

- Test of 2D detector and calibration next year.

NDP – neutron depth profiling spectrometer (results and collaboration projects)

- Continuation of cooperation with other faculties on lithium salts and cooperation on solid-state batteries.

MÖSSBAUER SPECTROSCOPY – in-beam Mossbauer facility (external funding and industrial partnership)

- In the future, the number of Mossbauer nuclei used for industrial experiments will be increased by producing them in the neutron beam.

FISH – a new multi-purpose neutron imaging facility

The current neutron imaging projects mainly run as educational and explorative projects on a test beam line. In 2016 the current temporary setup for neutron imaging at the test beam line will be enhanced, allowing for collaborations with external partners, keeping momentum in preparation for the actual realisation of FISH. In parallel, the detailed design of FISH will be initiated.

For a detailed planning see p. 31

FlexBeFa – a combined and flexible irradiation facility (external funding, design phase and initial mathematical simulations)

- Building and installation of prototype
- Tests using polymeric microspheres expected in 2016

ROG upgrade – Reflectometer for surface and interface research (planned)

- Half 2016 – half 2019

First experiments with PEARL

The new neutron powder diffractometer PEARL improves energy material research. PEARL will unravel the crystal structure of materials, for example energy materials such as batteries and hydrogen storage materials. Due to a new smart design, PEARL can be compared to the best neutron powder diffractometers in the world, even those with a neutron source ten times as powerful. The closest neutron diffraction facilities are located in Oxford, Paris and Grenoble.

PEARL is a unique instrument in the Netherlands and will also be used by researchers outside TU Delft.

Materials research has led to significant technological progress in the last few decades. Society now needs technological solutions for renewable energy, and new storage and conversion materials must play an important role in these solutions. PEARL has been constructed in order to be able to understand and develop these types of materials.

Calibrating, testing and opening workshop in 2015

The PEARL team calibrated and tested the instrument in the first half of 2015. Instrument performance was tested with new battery and magnetocaloric materials from TU Delft, new catalysts from TU Eindhoven, and thermoelectrics from the University of Groningen. During the summer stop of the reactor, shielding was improved, upstream of the sample. In the 4th quarter of 2015 further signal-to-noise improvements were achieved by installing a vacuum vessel at the sample position.

The opening of PEARL was celebrated on 24 September 2015 with a successful, scientific workshop given by and for 70 future users from various fields of research. Through a video link with the reactor hall the attendees were able to view how the sample in PEARL was bombarded with neutrons from the reactor.

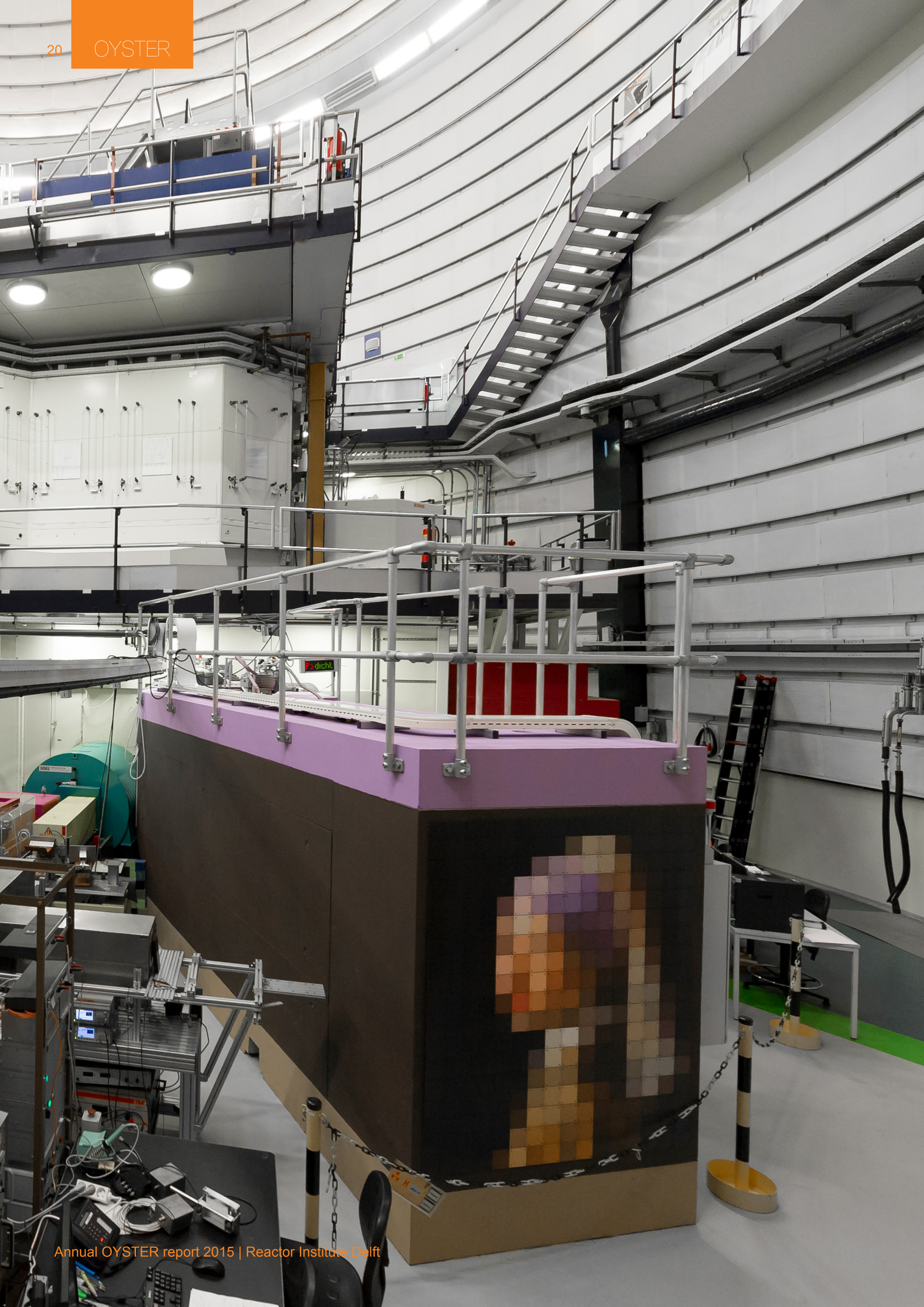
Atomic resolution

PEARL is very well suited to accurately locate lithium in batteries. The atomic resolution of PEARL will be used for storage materials for hydrogen and ammonia, and magnetocaloric material for energy conversion. Neutron diffraction is an important technique for studying the atomic structure deep underneath the surface of materials and is very sensitive to elements such as hydrogen and lithium in energy materials. The technique is also suited to fields of research such as zeolites, thermoelectrics and ferroelectrics, and fixed material solar cells.

Growing user base in 2016

The focus for PEARL in 2016 will be on ‘ producing science’. In-house collaborations on solid electrolytes, in-situ battery experiments and electrode materials and magnetocalorics are put in place, as well as nuclear materials. The first experiments with colleagues from the TU Delft faculties Mechanical Engineering (steels) and Chemical Engineering (perovskite solar cell materials) will extend the user base, as well as several collaborations with other universities in and outside the Netherlands. With a growing user base, the need for more support, sample environment and easier access can be expected and the required infrastructure should be developed in 2016 too.

Photo: Left: More than 5000 of these fiber ends were used in PEARL.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Tribute to Dr. Hugo M. Rietveld

The chairman of the Netherlands Organisation for Scientific Research (NWO) Prof. Jos Engelen, paid tribute to Dr. Hugo M. Rietveld during the opening workshop of PEARL on 24 September 2015 in recognition of his global scientific contribution to crystallography. The Rietveld Refinement method is used every day by hundreds of scientists to analyse powder diffraction data – generated for instance by PEARL – and to translate this data into the 3D atomic crystal structure of materials. Insight into the structure at this atomic level allows new batteries, fuel cells and related items to be designed.

Dr. Hugo Rietveld (84) is one of the most prominent crystallographers of the 20th century.

Photos: Left: PEARL in the reactor hall. Above: The chairman of the Netherlands Organisation for Scientific Research (NWO) Prof. Jos Engelen, paid tribute to Dr. Hugo M. Rietveld.

PEARL opens the doors to new storage and conversion materials.

Powder diffractometer PEARL reveals the fingerprints of atomic structures

The neutron powder diffractometer PEARL opened at the Reactor Institute Delft in 2015, is literally a pearl amongst diffractometers. “In terms of the visualisation of atomic structures, PEARL is absolutely world class”, according to physicist, Dr Lambert van Eijck, one of the founders of PEARL.

Materials research and technological progress go hand in hand. However, the solutions to producing better materials are becoming increasingly more complex. Neutron diffraction may play a key role in this, as it allows crystal structures of elements - which would otherwise not be visible - to be studied at an atomic level. “This enables a fingerprint, as it were, to be made of how atoms are arranged”, says Lambert.

Sustainable energy

PEARL is particularly sensitive to elements such as hydrogen and lithium which are present in energy materials. There is a global interest in this, as there is a great need for technological

solutions to sustainable energy with a focus on new storage and conversion materials. “We want to be able to store solar and wind energy, but in order to do this we need to know how lithium behaves in the storage materials. There is also a demand for better batteries for electric cars, so we need to properly understand the behaviour of lithium in batteries. And in order to continue developing magnetic cooling, we need to be able to thoroughly investigate the behaviour of magnetocaloric material”.

There is a significant - albeit indirect - social benefit to PEARL. According to Lambert: “PEARL is part of the infrastructure required to provide a contribution to the solution of current global energy problems”. Thanks to PEARL we are able to research energy materials. This should provide a basis to enable the successful development of energy storage facilities for, for instance, wind and solar energy, and to realise clever cooling methods, such as magnetic cooling. In addition to this PEARL can be used by researchers around the globe. Every scientist with experience of x-ray diffraction will know within thirty minutes how our neutron diffractometer works”.

Smart design

There is a significant opportunity for use by external parties given that PEARL can take on the competition with the best diffractometers in the world even though Delft has a ten times weaker neutron source. The foundation for this is a smart design co-developed with an Australian colleague. According to Lambert: “To put it simply, we have an instrument which due to an optimum combination of all the parameters is able to measure very rapidly, despite the fact of a small reactor. We can analyse a sample in an hour where that could take a day for some other diffractometers”.

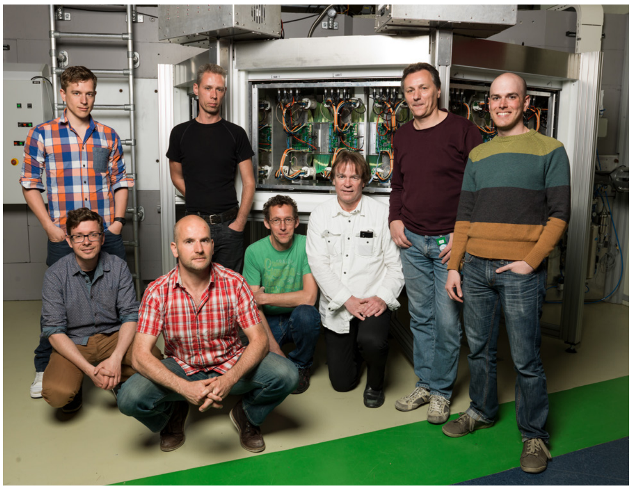


Photo: PEARL-team
(Herman Kempers Fotografie)

Positron Annihilation Lifetime Spectroscopy - POSH-PALS

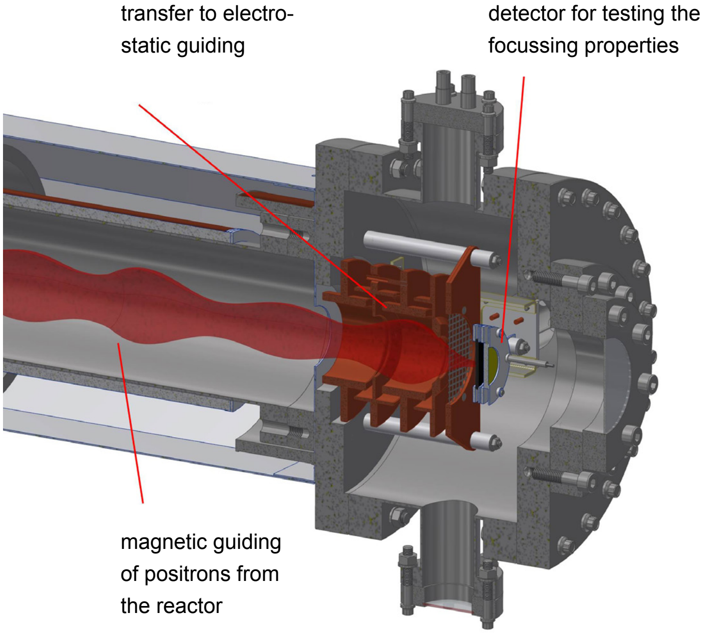
Microscopic defects play an important role in the physical properties of materials. For example, the presence of defects significantly affects the performance of promising thin-film amorphous silicon solar cells and plays an intriguing role in the recently discovered new class of highly-efficient perovskite solar cells. These defects, ranging from atomic vacancies to nano-voids, are usually too small to be made visible, e.g. with electron microscopy. Furthermore, their concentrations are often too low to be detected by standard methods. Positron Annihilation Lifetime Spectroscopy (PALS) is a unique method for identifying these defects and their concentrations in materials. Using PALS will help to understand how atomic defects are created and behave in solar-cell materials which is very important for developing production methods for improved solar cells.

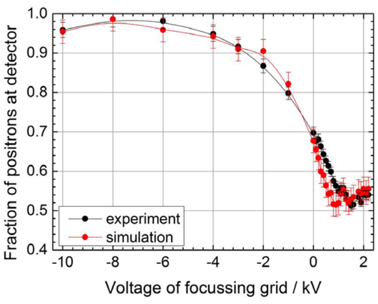
In the framework of the ADEM (A green Deal in Energy Materials) programme, the proposal “Thin-film positron annihilation lifetime spectrometer POSH-PALS for advanced characterization of defects and nanostructures of thin film solar cell layers” was approved in 2014. In 2015, a detailed design of the POSH-PALS spectrometer was developed based on computer simulation studies for beam transport. A main component is the timing system of the spectrometer: The positron annihilation lifetime is determined by the time difference between implantation and annihilation of positrons in the sample. In the POSH-

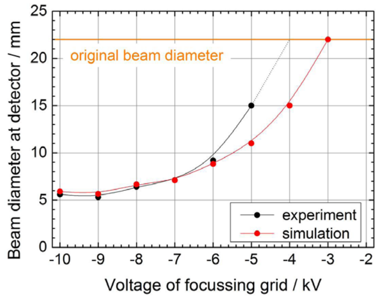
PALS spectrometer, the starting signal is determined by secondary electrons that are generated by the positrons passing through a carbon foil before entering the sample material. Detailed simulations helped in constructing and optimising the timing system. In addition, the essential parts for focussing the beam down to a sub-millimetre diameter have been installed and tested successfully.

As of the beginning of February 2016 scientists will install a new test chamber which was designed based on the simulations and experiments in 2015. The new chamber will be used to test and further optimise the timing system and the subsequent beam transport elements. It is expected that the first PALS measurements will be performed on real samples by the end of 2016. With an expected depth profiling range from a few nanometres to several microns below the sample surface and a time resolution of about 150 ps, the POSH-PALS spectrometer will be excellently suited for revealing the behaviour of defects in thin-film solar cells.

Since PALS is a non-destructive method, it is also well suited for investigating effects of innovative manufacturing steps introduced in the solar cell production. For example, recent research shows that the addition of hydrogen during the manufacturing process improves the long-term stability of solar cells, and our positron studies indicate that hydrogen reduces the mobility and growth of vacancy clusters. With POSH-PALS we will be able to investigate such effects in full detail, aiming to improve the stability and performance of solar cells. In addition, the flexibility of the POSH-PALS spectrometer enables studies of materials with applications in fusion and fission reactors and free volume in polymers.







The small-angle neutron scattering instrument - SANS

Photo: The SANS instrument

The small-angle neutron scattering instrument (SANS) allows the user to investigate structures with particle sizes from 1 up to a 100 nanometers. For larger particles neutrons scatter under smaller angles. This allows for a direct characterization of the particle size distribution within the material. Using this technique proteins, micelles, polymers, porous media and precipitates are typically investigated. These are of interest for the development of new products in the field of polymer science, colloids, emulsions, food science and metal alloys.

In 2015, the SANS instrumentation reached further completion ahead of the reactor modification reactor and installation of the cold source. In 2016 the 2D detector will be tested and the instrument calibration will be performed using the thermal neutrons of the current reactor source. The instrument offers the opportunity in the period prior to the cold source installation to study strongly scattering samples and enable preliminary neutron imaging studies.

Neutron Depth Profiling – NDP

Li-ion batteries are used extensively in smartphones and cars. With the help of neutrons from the reactor and a technology known as Neutron Depth Profiling (NDP), our scientists now know exactly how the lithium ions in these batteries behave during actual use.

In 2014, the battery researchers identified the charge transport process that determines the charging rate in a specific type of battery electrode, and over the previous year they have been testing various types of electrodes. This has helped our researchers to better understand how lithium ions are transported through electrodes. At the same time, they are working on Li-ion transport models that can be directly compared with the measurements.

Cooperation with other faculties

RID is cooperating with a number of faculties on NDP research. TU Delft's Aerospace Engineering (AE) and Mechanical, Maritime and Materials Engineering (3mE) faculties want to know how lithium salts are distributed in aircraft coatings. The lithium salts give the coating self-repairing properties; however, it is not known how mobile these salts are. As this process closely resembles how lithium is transported in Li-ion electrodes, our researchers are conducting joint experiments with these faculties to learn more about it.

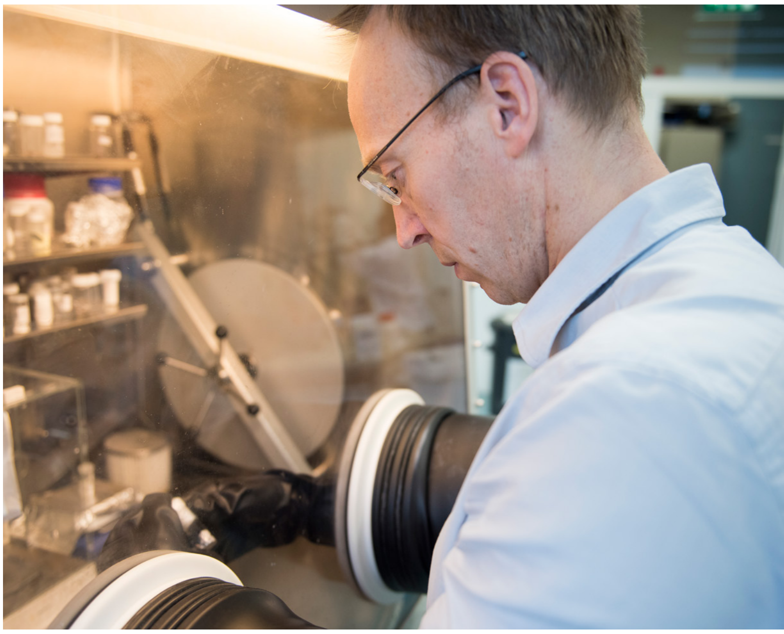


Photo: Associated professor
Dr.Ir. Marnix Wagemaker

Cooperation on solid-state batteries

During the spring of 2015, RID was paid a visit by Dr Richard D. Robinson of Cornell University. He is using NDP to learn more about the charge transport mechanism in a new type of electrode he has developed. There has been a lot of professional interest in his research since he published his first article on the subject. Discussions are underway between several universities and businesses on possible cooperation with a focus on solid-state batteries. In these Li-ion batteries, the liquid electrolyte is replaced by a solid form of electrolyte, which makes the battery much safer to use and could potentially lead to a higher energy density. However, the Li-ion transport is a limiting factor. NDP can be used to pinpoint exactly where the problems occur. Practical solutions are currently being sought to be able to measure these solid-state batteries using NDP.

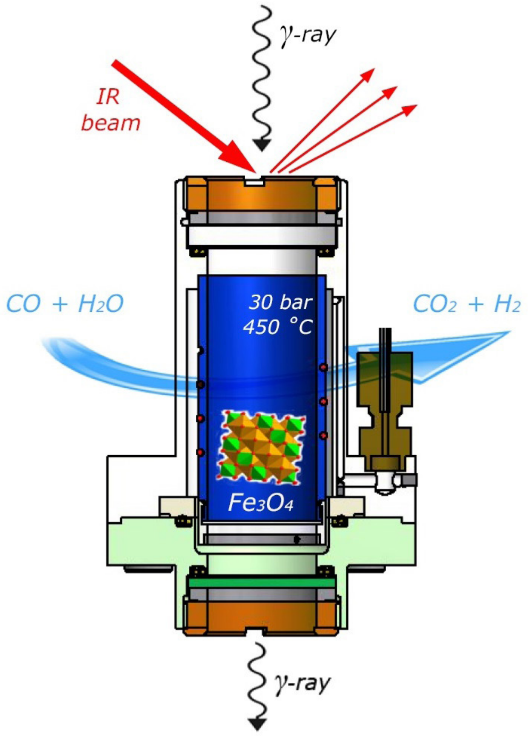
MÖSSBAUER

SPECTROSCOPY

The unique facility for combined Mössbauer/infrared spectroscopy, recently developed for the study of heterogeneous catalysts in their working state, has already attracted strong interest from industry partners. A collaboration project with Johnson Matthey Catalysts received financing within the Innovation Fund Chemistry (NWO) for the research and development of new water-gas shift (WGS) catalysts. The researchers will develop innovative chrome-free catalysts for the production of hydrogen. The research contributes to one of the main goals of sustainable chemistry, which is to develop technology that eliminates the use of hazardous chemicals in chemical production processes.

The WGS reaction has been a key step in hydrogen production since the early 1940s. Iron–chromium oxide is utilized as a primary catalyst in the WGS process because of its reasonable degree of activity and durability in most applications. The use of a chromium promoter is well established for commercial use in catalysts employed in the high temperature phase of the WGS reaction. However, chromium in its hexavalent form is a known human carcinogen. Furthermore it emits a toxic mist at elevated temperatures and is leached from the catalyst by condensed steam or cold water, which could be a threat to the environment, even in very small quantities. The proposed research, using the Mössbauer/IR spectroscopic facility, aims to study the iron oxides in WGS catalysts under industrial conditions and to clearly elucidate the effect of the chromium promoter on the morphology of the active site. The newly acquired knowledge on the role of chromium under WGS reaction conditions will be used to propose and test alternative less hazardous promoters with similar influence on the active phase of the catalysts.

In the future, the number of Mössbauer nuclei used for industrial experiments will be increased by producing them in the neutron beam. Neutron-capture prompt-gamma nuclei for in-beam excitation and continuously activated prompt-gamma Mössbauer nuclei will be used in catalysis, high-temperature superconductors, magnetic layers and nuclear waste research studies.



First Imaging Station Holland – FISH

Neutron imaging is a very versatile technique to reveal secrets inside materials without the need to destroy the object. Water or air bubbles inside hardening concrete or plastic parts trapped in steel housing can be viewed with neutron imaging, whereas this is not possible with X-ray imaging. FISH refers to First Imaging Station Holland and is a neutron imaging station that will be unique within a 500 km radius from Delft. This instrument is designed for neutron radiography and tomography, which given the nature of neutron radiation is complementary to X-ray imaging.

In 2015 a NWO Groot proposal was submitted to find funding for FISH. As part of the preparations for this proposal a workshop was organised for potential users together with international experts in the field to focus on the wishes of the Dutch user community. This resulted in a good overview of the science case which is essential to realising a successful neutron imaging instrument. At the end of 2015 a postdoc started work on different topics concerning FISH. His role - apart from the continuous search for funding – will also be to strengthen the user community with more pilot experiments.

The current neutron imaging projects mainly run as educational and explorative projects on a test beam line. In 2016 the current temporary setup for neutron imaging at the test beam line will be enhanced, allowing for collaborations with external partners, keeping momentum in preparation for the actual realisation of FISH. In parallel, the detailed design of FISH will be initiated.

- January 2016: Improvement of current test beam line (beam line brightness and resolution and detector resolution).
- January-December 2016: Simulation/calculation FISH optics and shielding;
- February 2016: Neutron imaging on Interface Clay/Concrete; collaboration with faculty of Civil Engineering and COVRA; MSc end project.
- April 2016: Plant root growth tomography in collaboration with Wageningen Agricultural University; BSc end project.
- May June 2016: First explorations with industrial partners
- September-December 2016: Cultural Heritage explorations with Boerhaave museum Leiden.

Photo:
Assistant professor
Dr.ir. Antonia
Denkova

Irradiation facilities

Combined and flexible irradiation Facility (FlexBeFa)

Radiation therapy with holmium microspheres is expected to become a major procedure for the treatment of liver tumors. Microspheres for the UMC Utrecht have been irradiated at Delft for a few years now. Patients with liver cancer are being treated with these irradiated holmium microspheres at the Utrecht hospital. The challenge for the Delft researchers is to deliver microspheres which will be more radioactive, i.e. will contain more 166Ho.

In 2015 a decision was taken to combine the Shielded Irradiation Pneumatic Facility (SIPF), the Resonance Irradiation Facility (RIF) and the Gamma Irradiation Facility (GIF) into one facility providing all three applications: the FlexBeFa. A facility is envisioned in which the exposure to neutrons and gamma-rays can be tuned by positioning shields of various composition in a modular way.

Dr Ir. Antonia Denkova: "The new facility will deliver microspheres that will be more radioactive, i.e. will contain more 166Ho, which will help to improve therapy but also to treat patients outside the Netherlands.

The orientation phase focused in 2015 on the design of this flexible facility supported by initial mathematical simulations. In line with the original SIPF plan, the impact of lead shields was initially studied applied as modular and easily exchangeable parts. Computer simulations indicated that a desired reduction of the gamma-ray dose could be achieved while maintaining the neutron flux.

More 166Ho radioactivity in polymeric microspheres to treat liver cancer

The new facility will provide an opportunity for reducing radiation damage to, for instance, 166Ho polymeric microspheres exposed to extended irradiation times. This is needed to achieve higher specific activity. In a facility such as this the samples can be hydraulically transported in and out of the irradiation position. The hydraulic mechanism also allows for additional cooling of the samples during irradiation. In 2016, we plan to have built and installed a prototype of the flexible facility and to test it using the polymeric microspheres.

'Technologiestichting' STW subsidy

A research proposal for additional funding for the design, implementation and testing of such a facility was submitted to, and granted by the Technology Foundation STW. This project is a collaboration between the TU Delft and UMC Utrecht and supported by industrial partners (NRG, Quirem and Mo989).

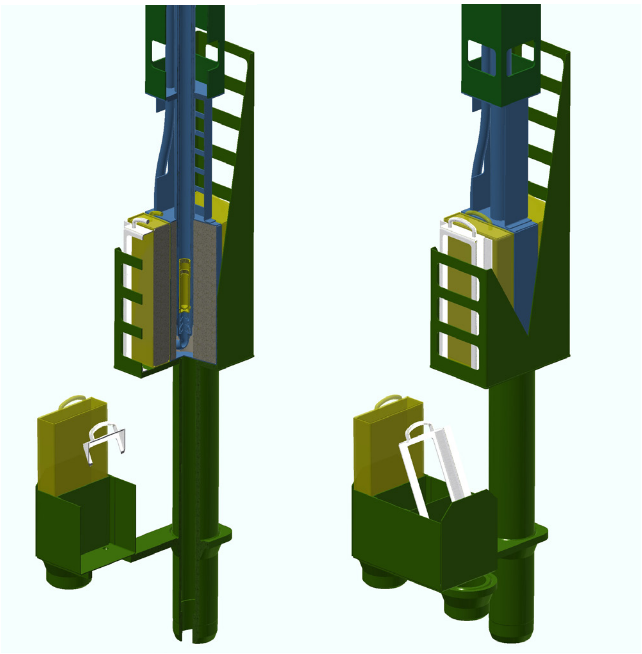


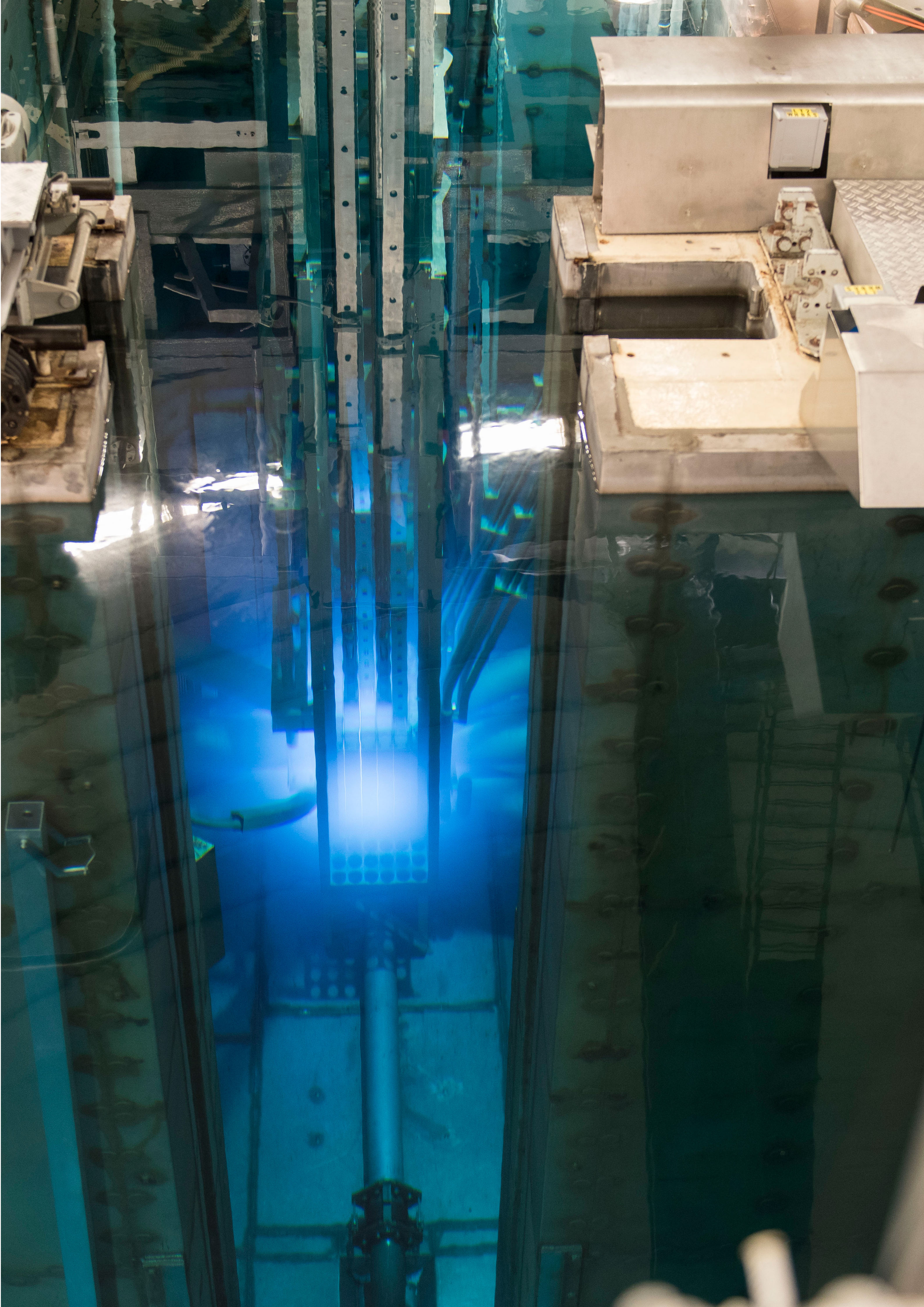
Figure 1. The FlexBeFA facility. On the left: cross-section of the facility, and on the right: the complete facility. The grey colour represents the shields, for instance lead, and the yellow colour simply indicates the alumina coating. The facility is composed of three parts which can be stored under water in the storage box at the bottom of the facility, if not needed.

Faster and cleaner Mo99 production route

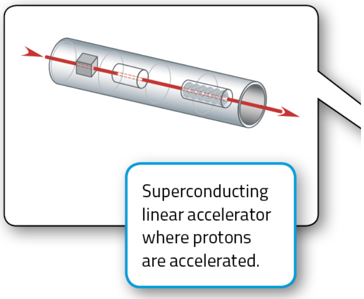
Medical isotopes are used for tumour diagnostics. One of the favorite isotopes is technetium that can be extracted after decay of molybdenum-99. Molybdenum is produced by fission of uranium and can be extracted from fuel plates after irradiation for a month in a high flux reactor. The molybdenum is loaded in so-called ‘generators’ and transported to hospitals. This production process leads to a relatively large waste stream.

Research is being undertaken in the framework of OYSTER into a new production route of molybdenum by irradiating a solution of uranyl-sulphate in a beam tube of the reactor. As the molybdenum is produced in a fluid, it can most probably be extracted faster and cleaner than through existing methods. This approach could lead to about 2% of global demand being produced in Delft. The process could be adapted for large scale production in the High Flux Reactor in Petten or PALLAS after the research and demonstration phases.

In 2015, research was undertaken into the safe removal of heat produced in the facility as a result of fission and gamma ray interaction with the structural materials. In the framework of a collaboration between research groups, a PhD research was started, with the aim to efficiently remove the molybdenum from the uranium-sulphate solution.



The European Spallation Source (ESS)

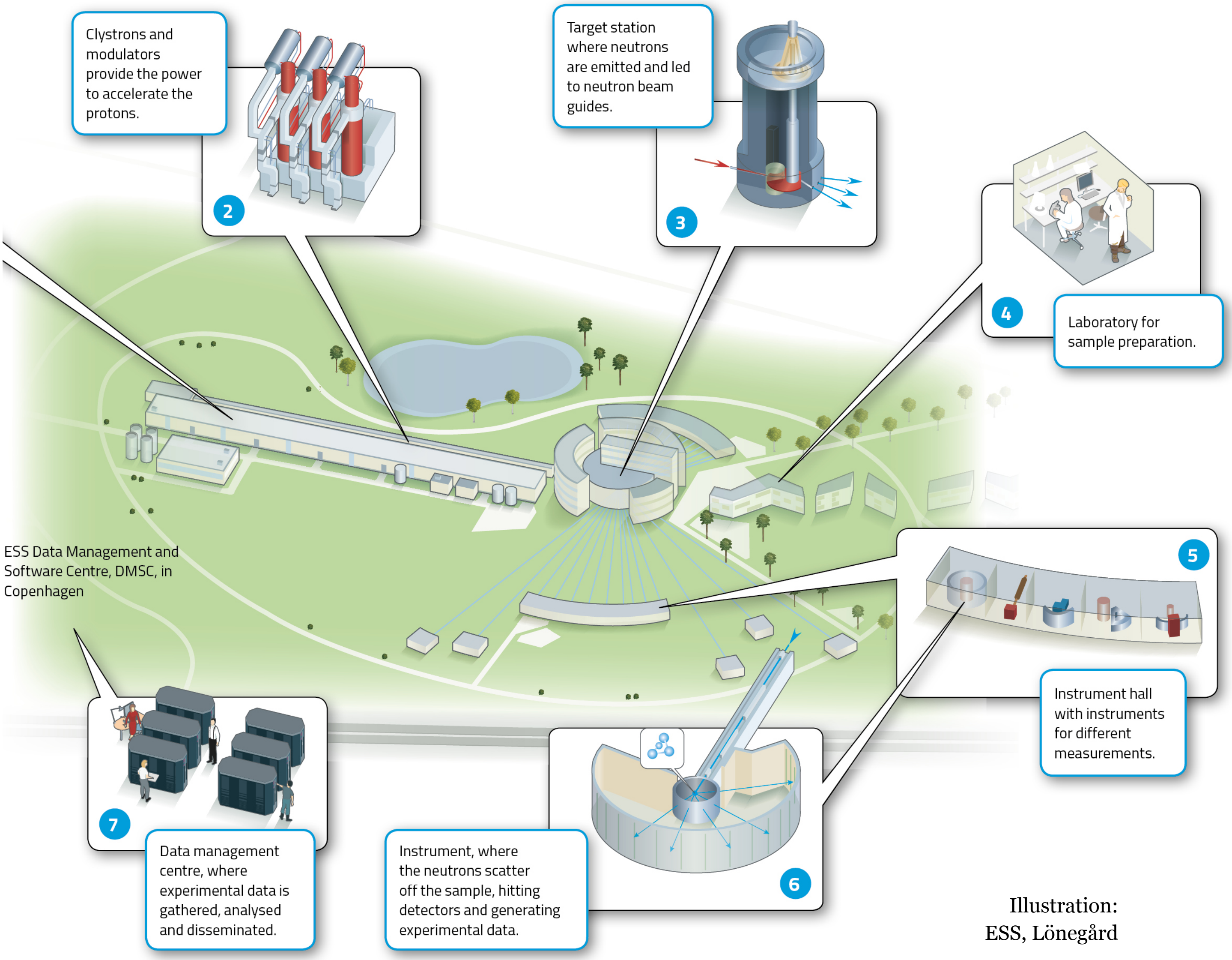


The European Spallation Source (ESS) is a European large scale facility being built in Lund (Sweden). It will be a neutron source including a suite of instruments, where neutrons are used to study all types of materials and gain knowledge about their structure and function.ESS will be the world's premier neutron source for science and will play a strategic role in maintaining and improving the position of Europe and its competitiveness among the knowledge-driven economies. In the Netherlands, many research activities would gain great benefit from participation in ESS, especially in the fields of life sciences (with food and plant sciences), engineering materials, magnetism, nanotechnology, cultural heritage, and chemistry.

The RID is the Dutch representative for the ESS and contributes with its unique expertise in the field of neutron techniques to the development of instruments for the ESS. Through the OYSTER project RID is able to invest in the ESS pre-construction program with four RID developmental work packages.

Because of the (financial) size of the ESS project an international approach is needed and many European countries have already partnered up with the host countries Sweden and Denmark. The Netherlands has also expressed its interest, but has not made a formal financial commitment yet and, as a result, has the official status of observer.

RID took the initiative in 2015 to propose the European Spallation Source (ESS) for the Roadmap Large-Scale Research Infrastructure. The Dutch scientific and industrial community will strongly benefit from a partnership-based access to ESS in maintaining and strengthening the position and innovative competitiveness of the Netherlands among the knowledge-driven economies. The importance of ESS for



Clystrons and modulators provide the power to accelerate the protons.

Target station where neutrons are emitted and led to neutron beam guides.

Laboratory for sample preparation.

ESS Data Management and Software Centre, DMSC, in Copenhagen

Instrument hall with instruments for different measurements.

Data management centre, where experimental data is gathered, analysed and disseminated.

Instrument, where the neutrons scatter off the sample, hitting detectors and generating experimental data.

Illustration: ESS, Lönegård

the Netherlands has largely been recognized by the materials research community. Four universities have been actively involved in the proposal (TU Delft, TU/e, RUG & WUR) and the proposal received ample support from industry, both large companies as well as SME’s. Mid 2016, the roadmap will be finalized by the permanent committee Large-Scale Infrastructure.

Photo: ESS

Review by NWO

Every year, the “NWO OYSTER Advisory Committee” reviews the progress and implementation of the OYSTER project and in June 2015 the team of external experts visited RID for the 3rd time. The team of external experts consists of the following people:

> *The committee: ‘OYSTER has made huge progress during 2015 and is becoming ONE common project managed as a real construction project.’*

Prof. K.N. Clausen
Head of the Neutron and Muon Research Department and vice Director of the Paul Scherrer Institute (Switzerland)

Prof.dr. J.F. Verzijlbergen
Head of the Department of Nuclear Medicine at the Erasmus MC

Dr. Dimitri N. Argyriou
Director for Science at the European Spallation Source

Dr. Nico Kos
Senior Manager (International) Programme Innovation at the Chemical & Physical Sciences Division of the Netherlands Organisation For Scientific Research (NWO)

Comments/Recommendations	Implementation/Solutions
The funding for scientific and/or user support of the instruments will not match the standards of facilities with a substantial user programme. This is a capacity issue that will become increasingly important and needs attention as the different facilities start to be ready for science.	The capacity issue related to the accommodation of users has our attention. The user issue is being addressed thoroughly with the commissioning of PEARL: the PEARL users approach is to serve as a blue-print for the instruments that are still being considered and/or in development.
The instruments/experiment facilities are being viewed more as small individual projects within the big OYSTER project and no one has as yet taken full ownership.	"Owners" are assigned for all the instruments/facilities. The challenge is to raise additional funding, and schedule both development and user accommodation.
The most critical issue for the reactor modification works will be a delay due to undefined licensing procedures or legislation leading to unexpected new technical or operational requirements. This is a real issue that, in spite of supportive regulators and good relations with the contractor, could lead to a serious budget overrun.	The TU Delft Executive Board is aware of this issue. It has RID's continuous attention also in consultation with the contractor and regulator ANVS.

Specific technical recommendations:

- Consider having a technical review before going into the final design of the cold source. - Consider a radial collimator for PEARL.	- The expert team members were intensively involved in the design of the cold neutron source and played an important role in the final lay-out of this equipment. - The radial collimator is still under consideration. We have reached an acceptable background level without, using a vacuum vessel at the sample position. Current in-house research is done at room temperature.

Financial overview

The KHC consortium will be able to prepare the final cost proposal for the implementation of the DSR related works as a result of the final revision of the Dutch Safety Requirements (DSR) being available in October 2015 and the selection of subcontracting parties in December 2015. Along with the price for the original scope of work as described in the agreement of 2014, a final price for the total costs for the reactor modification works can be made and presented to TU Delft. A Go/NoGo decision will be taken by the Executive Board after this. These prices will be provisional and therefore the continuation of the reactor modification depends on the acceptance of the final KHC consortium price by the Board.

The available OYSTER project budget amounts to €117 million, covering the initial investments as well as the basic reactor-associated operational costs over a period of 10 years. In 2012 the Dutch government awarded €38 million for OYSTER and TU Delft contributes €74 million in kind. Furthermore, TU Delft stands surety for an additional €5 million. This is part of the co-funding (industrial, scientific etc.) that has to be added to RID/OYSTER to fund the development, commissioning and exploitation of instruments and facilities over the total 10-year OYSTER program period. The additional costs for implementing the new Dutch legislation DSR are not known yet and will be added to the original budget.

In 2015, a total of €4.6 million was spent on the license process, basic engineering, the positioning of the OYSTER facility, and Dutch Safety Requirements (DSR). TU Delft contributed €8.6 million in kind to the OYSTER project in 2015.

The PEARL project was completed in 2015. The total €1.3 million invested was covered by the OYSTER investment and the in kind contribution from TU Delft. Plans are being formulated for the further realisation of instruments and irradiation facilities in order to attract external funding (e.g. for FISH, Mössbauer and Irradiation Facilities). The funding for SANS is being continued.

Communication

The communication strategy in order to share and instil the OYSTER key message to the relevant stakeholders and research communities was continued. We want to inform the various groups about the facilities, instruments, opportunities and know-how available at the Reactor Institute Delft.

In 2015 this resulted in, amongst others:

- A scientific opening workshop for the first neutron instrument PEARL for 70 future users from various fields of research. The chairman of the Netherlands Organisation for Scientific Research (NWO) Prof. Jos Engelen paid tribute to Dr. Hugo M. Rietveld in recognition of his global scientific contribution to crystallography.
- Development of an online proposal system where researchers can easily submit a research proposal to request for PEARL-beamtime, and in the future also for other instruments. The first version of the proposal system is accessible via our PEARL page on our website.
- A new compact brochure about OYSTER (in English and Dutch) for visitors of the RID and all other interested parties.
- An update of the Dutch OYSTER website where the general public can find information about the programme. An English version of the website has been added.
- A new internal online news portal which makes is possible for employees to share their news about OYSTER and projects they are working on.
- Celebrating successes like PEARL and increasing research funding in Europe among all employees.
- Tours in the reactor hall and experiment hall for various interested parties.
- As the Dutch contact-point for the ESS involvement of the RID in the European BrightnESS and SINE2020 projects. With these programmes collaboration at European level in expanding the neutron user community will be intensified in 2016. Through OYSTER an excellent gateway for the Dutch neutron user group of researchers and companies who want to work with the ESS will be realised.

See: http://www.tnw.tudelft.nl/en/cooperation/facilities/reactor-instituut-delft/pearl-new-neutron-diffractometer/

BrightnESS is a European Union-funded project within the European Commission’s Horizon 2020 Research and Innovation programme in support of the European Spallation Source (ESS).

SINE2020: Science and Innovation with Neutrons in Europe in 2020. This consortium of 18 partner institutions is funded by the European Union through Horizon2020.

Planning

The latest revision of the 2014 project schedule refers to the completion of the OYSTER reactor modification by Q1 2018. One of the aspects which will affect the project schedule is the new legislation Dutch Safety Requirements (DSR) for new and existing nuclear facilities. The final version of the DSR will be part of the legal basis for the contract agreement between the KHC consortium and TU Delft. This will incorporate the financial and schedule impact following the implementation of the DSR.

In December 2015 an important decision was made by the KHC consortium with the selection of Royal Haskoning DHV (RHDHV) as subcontractor for the implementation of the CNS Utility works. Furthermore, the contractor for the CNS In-pile component was selected and KHC will be able to prepare a construction schedule based on the availability of the final DSR and the input from contracting parties who will implement the site works. Based on the present situation, it is anticipated that the OYSTER project will be (probably) closed out at the end of 2018.

Activity name
TOTAL INSTALLED COST OVERALL ESTIMATE
CONSTRUCTION SECURITY CHECK
SHUT-DOWN REACTOR
FABRICATION EQUIPMENT REACTOR MODIFICATIONS
START-UP REACTOR
CLOSE-OUT OYSTER PROJECT
GENERAL
TIC OVERALL ESTIMATE
CONSTRUCTION SCHEDULE
CONSTRUCTION SECURITY CHECK
REACTOR MODIFICATIONS
DUTCH SAFETY REQUIREMENTS DEFINITION
'MILIEUEFFECTRAPPORTAGE' PREPARATION + REVIEW
SAFETY REPORT (SAR) PREPARATION + REVIEW
LICENSING PROCEDURE
BUILDING PERMIT
OBJECTION PROCEDURE
BASIC ENGINEERING REACTOR MODIFICATIONS
DETAILED ENGINEERING REACTOR MODIFICATIONS
FABRICATION EQUIPMENT REACTOR MODIFICATIONS
SHUT-DOWN REACTOR AND DISMANTLING CORE
DRAINING REACTOR BASSIN 1ST STAGE INCL. DISMANTLING HANGER ETC.
AS-BUILD CHECK CORE AND BASSIN CONFIGURATION
DRAINING REACTOR BASSIN 2ND STAGE FOR BEAM TUBES DRY
DISMANTLING MIRROR GUIDE SYSTEM BEAM TUBE R2
MODIFICATIONS BEAM TUBE R2 AND REACTOR CORE
MODIFICATIONS REACTOR BASSIN
INSTALLATION HOTCELL, ISOTOPE - AND LOOPSYSTEM FACILITIES
REFILL REACTOR BASSIN 1ST STAGE INCL. INSTALLATION NEW CONTROL RODS
AS-BUILD 0-MEASURING AND REFILL REACTOR BASSIN 2ND STAGE
INSTALLATION INPILE PART
DETAILED ENGINEERING E&I MODIFICATIONS RELATED TO CNS INPILE PART
MODIFICATIONS E&I RELATED TO CNS INPILE PART
CNS UTILITIES CONTROL ROOM
DETAILED ENGINEERING CNS UTILITIES AND CONTROL ROOM
BUILDING PERMIT
INSTALLATION NEW CNS UTILITY BUILDING
DETAILED ENGINEERING CNS CRYOGENIC EQUIPMENT
INSTALLATION CNS CRYOGENIC EQUIPMENT
DETAILED ENGINEERING CNS E&I MODIFICATIONS
INSTALLATION PIPING AND E&I
START-UP / COMMISSIONING

Start	Finish	2014				2015				2016				2017				2018			
		Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4
	31/Dec/15								■												
	31/Dec/16												■								
	1/Sept/17															■					
	31/Mar/18																	■			
	1/Oct/18																				■
	31/Dec/18																				■
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1/Apr/16	31/Dec/16										■	■	■								
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1/Jul/15	31/Dec/16							■	■	■	■	■	■								
1/Jan/15	28/Feb/17					■	■	■	■	■	■	■	■	■							
1/Mar/17	1/Sept/17														■	■					
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Date	1 Dec 2014	31 Jan 2015	22 Apr 2016
Revision	Rev. 14	Rev. 15	Rev. 16