D4.1 – Selection of materials and components for experimental testing and test plan

FUEL CELLS AND HYDROGEN
JOINT UNDERTAKING

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Acronyms

Short Acronym	Description
3M	3M Company: U.S. American multinational conglomerate corporation (MEA producer)
PM	Particulate Matter
BoP	Balance of Plant (periphery components of a fuel cell stack for processing)
BPP	Bipolar Plate (core component of a fuel cell stack)
CCM	Catalyst Coated Membrane (core component of a fuel cell stack)
CO	Carbon monoxide
DI	De-Ionised (water) with electrical conductivity < 50 µS/cm²
EDX	Energy Dispersive X-ray spectroscopy
EIS	Electrochemical Impedance Spectroscopy
GDL	Gas diffusion layer (core component of a fuel cell stack)
H_2	Hydrogen
HX	Heat Exchanger
ICE	Internal Combustion Engine
ICF	International Code of Safety for Ship Using Gases or other Low-flashpoint Fuels published by the IMO
IMO	International Maritime Organization
MCFC	Molten Carbonate Fuel Cell
MEA	Membrane electrode assembly (core component of a fuel cell stack)
MFM	Mass flow measuring
MSC	MultiSingleCell screener stack concept developed by VTT
NaCl	Sodium Chloride, sea salt
NH_3	Ammonia
NO_x	Nitrogen Oxides (refers to NO, NO_2, but also N_2O_3 und N_2O_4)
OCV	Open Circuit Voltage
PAFC	Phosphoric Acid Fuel Cell
PCB	Printed Circular Board (electronic devices)
PLC	Programmable Logic Controller

PEMFC	Polymer Electrolyte membrane Fuel Cell
PGM	Platinum Group Metals i.e. ruthenium (Ru), rhodium (Rh), palladium (Pd), osmium (Os), iridium (Ir) and platinum (Pt)
Pt	Platinum
QA	Quality Assurance
SEM	Scanning Electron Microscopy
SOFC	Solid Oxide Fuel Cell
SO_x	Sulphur Oxides (refers to many types of sulphur and oxygen containing compounds such as SO, SO_2, SO_3, S_7O_2, S_6O_2, S_2O_2, etc.)
U/I	Polarisation curve: Voltage/Current
VOC	Volatile Organic Compounds (definition see Footnote 1)

Executive Summary

The e-SHyIPS project aims to define the new guidelines for an effective introduction of hydrogen in maritime passenger transport sector and to boost its adoption within the global and EU strategies for a clean and sustainable environment, towards the accomplishment of a zero-emission maritime transport Scenario. The goal of e-SHyIPS is to move from the idea to the application, filling the existing gaps in normative and technical knowledge concerning all the related aspects on hydrogen in the maritime transport sector. By means of an ecosystemic approach, e-SHyIPS proposes theoretical pre-normative research activities on standards, simulation and laboratory experiments, design of an appropriate certification process, spot future standardization activities to enhance the EU normative and regulatory landscape.

Hydrogen as a potential energy carrier is seen as an effective option for achieving global emission reduction targets and is also seen as part of the International Maritime Organization (IMO) strategy, although the legal framework for hydrogen-powered ships has not yet been developed. One of the most important tasks to meet the project objectives is the investigation of safety systems, materials and components, and bunkering procedures. The methodology is "ship independent" to avoid the burden of bespoke projects. In this deliverable, the activities of work package WP4 Task 4.1 were discussed in more detail. The scope and objectives of this Deliverable 4.1 as part of work package WP4-“Materials and component testing”, are the definition of test procedures and plans as well as the selection of the critical components to be tested to ensure safe and durable operation of fuel cell systems in maritime applications within the further course of the work package. Firstly, typical fuel cell components for the marine sector were compiled, as well as the main factors of environmental conditions that may lead to negative or critical effects. Based on this, fuel cell materials, components and appropriate test procedures were defined by which the mentioned stressors can be further investigated in order to avoid their detrimental effects on the fuel cell system. The resulting test plan serves as a starting point for further WP4 work. Based on the results of this WP4, it is planned to develop a pre-standardisation plan for updating the IGF code for hydrogen fuels for passenger ships and a roadmap for promoting the hydrogen economy in the maritime sector.

1. Introduction

1.1 Scope and Objectives

This report describes the activities of Tasks 4.2 and Task 4.3 of Work Package 4. As the overall objectives of this work package are primarily to provide technical knowledge to support discussions on the development of a coherent regulatory framework for the risk assessment and management of hydrogen on ships and to provide technical guidance on materials, components and the general design of piping for H_2 containment and handling, basic considerations are necessary at the beginning. In parallel, experimental validation of the main components of the hydrogen power train will be carried out to ensure that there are no impurities in the materials and components that could lead to degradation of the fuel cell system performance. The outcome of this work package will be a clear and practical guide that can be easily used in future efforts to install hydrogen fuel cell systems on board passengers' ships. The first step is to define the main components of a hydrogen propulsion system. Based on this definition, an appropriate selection of tests can then be carried out to ensure the suitability of the components described. This selection and definitions, as the main task of T4.2/T4.3, are the subject of the present work package deliverable D4.1.

1.2 Methodology

The following approach was chosen for the identification of the necessary investigations. First of all, it had to be worked out which system components are used in principle for the use of hydrogen fuel cells in maritime applications. In parallel, the influence of the expected environmental conditions on the individual components must be worked out. These environmental conditions naturally vary significantly from the intended area of application: in particular, harmful gases in the media supplied are a potential source of damage and/or reduction of the service life of fuel cells. On this basis, a matrix of the materials and components to be tested and the conditions to be applied has to be developed and described in test plans.

1.3 Connection with Other Deliverables

This D4.1 "Selection of materials and components for experimental testing and test plan" is related to T4.2 and T4.3 of WP4, and serves as input for:

- D4.2 "Interim test results from material and component testing and post-mortem Analysis", which is due in M24 (also related to the activities of T4.2/T4.3/T4.4)

and finally

- D4.3 "Test results from material and component testing and post-mortem Analysis", due in M39 as conclusion of the activities T4.2/T4.3/T4.4

Beside the direct links towards the actions in the same work package there are several crosslinks and implications to other work package WP 3: "Safety System Experiments" Especially the results gained in the tests will influence

- T3.3 "Safety Systems Definition and Preliminary Design" and also
- T3.4 "Safety Engineering / Risk Assessment / Risk management

Furthermore there is a connection towards WP1: "State of the Art & theoretical studies"

- T1.1 "Project concept and functional scenarios definition and review"
- T1.4 "State of the art of safety technical framework and safety plan

The information that have been collected in these work package, especially the definition of the scenarios, the decisions on the fuel and fuel cell types are the basis of the test plan developed and described in this Deliverable report.

Due to the close interlocking of the individual tasks and work packages, there may also be other connections and influences. These can eventually only be discovered during the test phase.

2. Determination of environmental influences

2.1 Scenarios of applications

In principle, the term "maritime application" refers to use in, on and under water. In most cases, however, ships are meant. In addition to the main drive train, auxiliary energy or the auxiliary consumers can also be supplied with electrical energy by a hydrogen fuel cell. However, the concept described here are just as valid for applications, e.g. in port facilities. An important distinction in the application of maritime systems lies in the differentiation between offshore shipping and inland shipping. In addition to the application of different legislative requirements, this also applies to the presence or absence of certain pollutants in the environment as well as the different requirements for sea and operational conditions. This has to be taken into account. To reach the widest impact and to obtain a pre-standard normative plan intended to apply to as many ships as possible, the project approach is vessel independent: this implies that the project does not even focus on a specific fuel cell type or technology, but rather on a global approach. In particular, the different fuel cell technologies differ somewhat from each other, so that more emphasis must be placed on some points. This is especially true for Polymer electrolyte membrane fuel cells, PEMFCs, which react much more sensitively to impurities from the environment than other fuel cell types. The following Figure 1 shows the basic structure of a typical fuel cell:

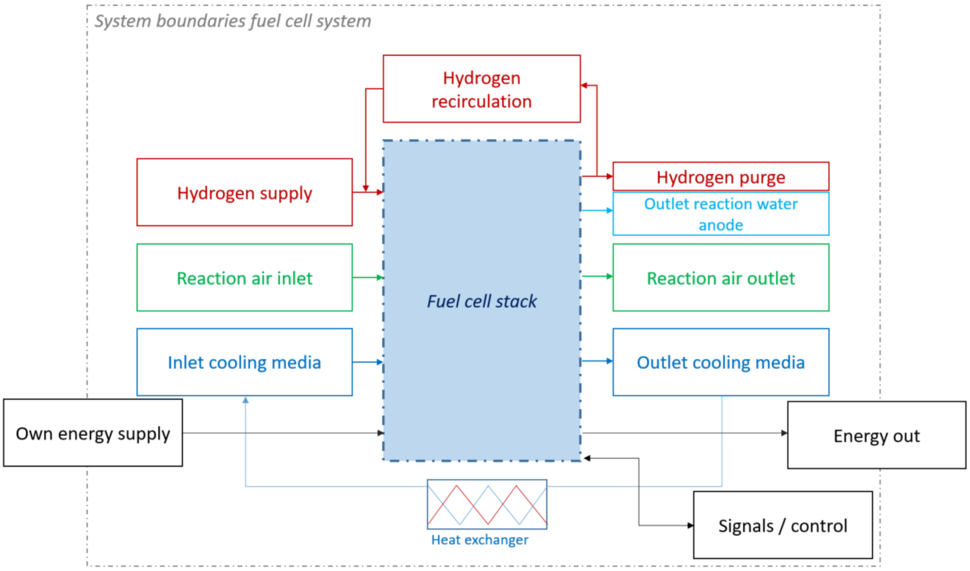

Figure 1 System boundaries schematic

A more detailed view is given in the P&I diagram in Figure 2:

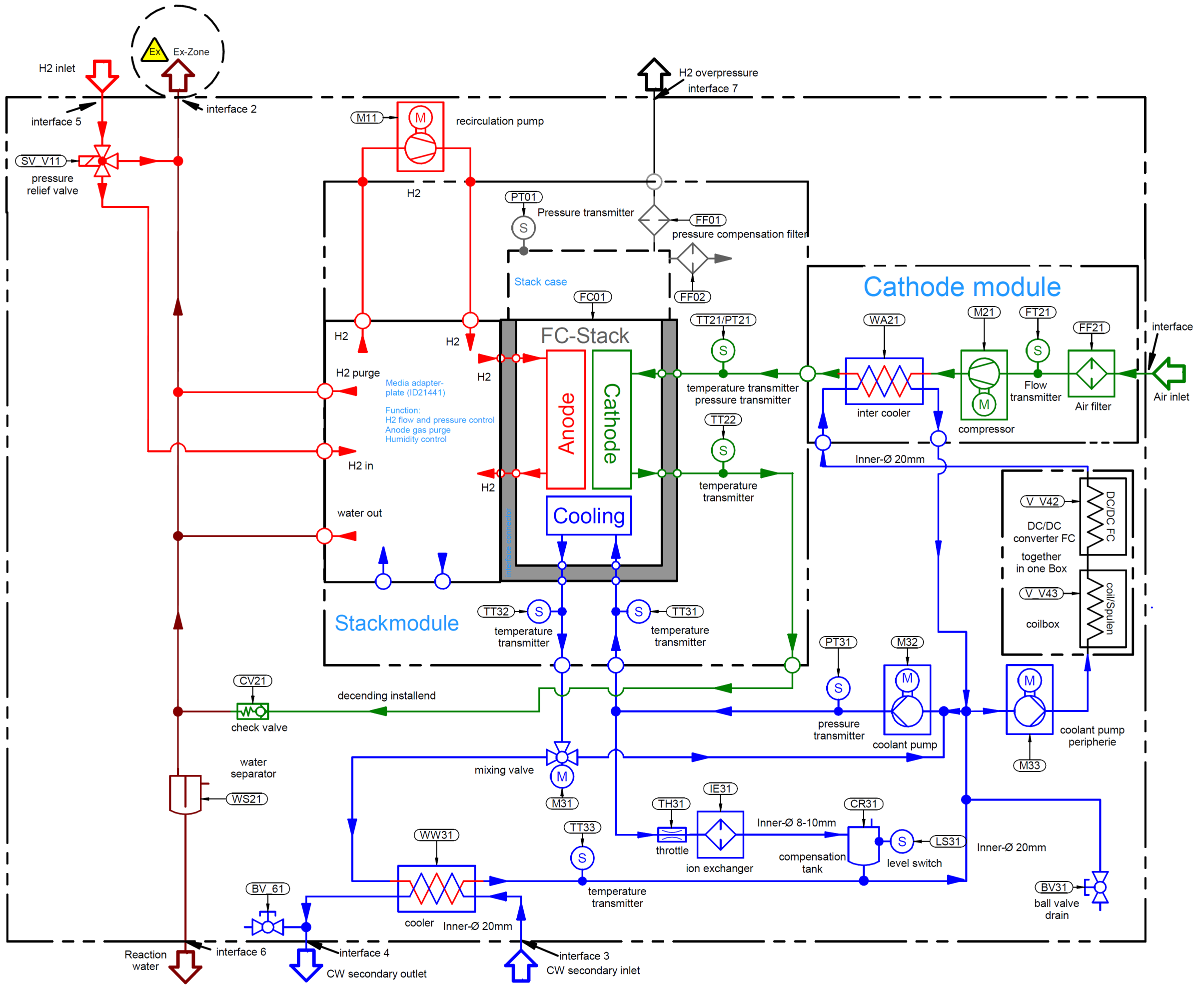

Figure 2 Generic architecture / P&I diagram of a fuel cell system

Within the framework of the e-SHyIPS project, three Scenarios (small, medium and large) have been built to be flexible and scalable to foster knowledge transfer in between the different experiments and to reach the expected project KPIs. They represent the operational profile and vessel design reference for the simulations and the laboratory experiments of WP2, WP3, WP4 and WP5, and they will support the creation of the roadmap for the boost of Hydrogen economy in the maritime passenger ferry sector in WP6. In order to adopt a coherent approach, WP4 will focus on the most stringent requirements and not on the individual characteristics of the respective class (Scenario). This means that the components and fuel cell systems used can then also be used in general.

2.2 Identification of potential pollutants

Pollutants in the media-carrying parts of a fuel cell can have a strong influence on the current performance (power output) as well as the service life. Most fuel cell systems with lower operating temperatures than 100 °C (such as the polymer electrolyte fuel cell, PEMFC) are extremely sensitive to this. Fuel cell types with higher operation temperatures such as PAFC (160 - 220 °C), MCFC (500 - 700 °C) or SOFC (450 - 1000 °C) are significantly less sensitive regarding those chemical compounds. This is referred to as reversible or irreversible degradation, depending on whether the effects of the pollutants can be eliminated by suitable operating strategies. The cooling loop, the hydrogen path (anode) and the reaction air path (cathode) should be mentioned as media-carrying circuits.

A) Cooling loop

The cooling circuit is usually designed as a closed system (at least a closed primary cooling circuit is common for the fuel cell. Further heat dissipation and/or utilisation of the waste heat can be achieved by decoupling a secondary circuit by means of a heat exchanger (HX) and is not relevant for this consideration). The design as a closed system means that no harmful foreign matter can enter the circuit, provided this has been taken into account in the design. Negative temperature influences can also be largely determined in advance. Depending on the ambient temperatures to be expected, the cooling medium must be selected accordingly. Ethylene glycol-based cooling media, which can tolerate temperatures down to -36 °C without any problems, are common.

B) Hydrogen path

Hydrogen is an essential starting point for this consideration. The hydrogen circuit must also be shielded against the intrusion of pollutants from the environment. This is a general requirement for efficient operation of a fuel cell. This leaves the requirement for the provision of hydrogen that meets the purity requirements of the respective fuel cell. However, this requirement can depend heavily on the technology used. For example, PEMFCs are much more sensitive to impurities (especially carbon monoxide, CO) than PAFCs, MCFCs and SOFCs (in that order). Provided that the purity requirements are met, no harmful influences from the environment can be expected in this cycle either.

C) Reaction air path

Especially in the air section harmful substances from the operating environment can make themselves felt. In contrast to the (closed) circuits described above, the air gap is open on both sides. Thus, especially during operation, harmful gases can also be sucked in by the reaction air. If the cathode system is not closed off after operation, substances can migrate into the cell through the air outlet even during standstill/non-operating phases. Contaminants may also permeate through the membrane to the anode.

The effects of fuel cell poisoning gases in the reaction air are well studied mostly understood and widely described in the literature [1]-[13] and there are also mitigation strategies described. [1] Hence they will be not examined in detail in scope of the e-

SHyIPS project. Since, according to Figure 2 the inlet of the air section is mostly equipped with an effective air filter unit, the contamination of the reaction air stands or falls with the quality of this filter. Therefore, this point is also emphasised in the tests described here. A short overview on typical air contaminants and the effects on a fuel cell (more specific, a PEMFC) is shown in Table 1.

Pollutant species	**Impact on fuel cells**	**Mode of effect**	**Remark**
VOC[1]	Mostly reversible	Hydrophilization of GDL and CCM adsorption and blocking of catalyst (concurring to oxygen reduction reaction)[12][13]	Aromatic compounds have stronger effects as aliphatic compounds
SO_2 (SO_x)	Very strong negative irreversible impact	Adsorption and blocking of catalyst (concurring to oxygen reduction reaction) Cross-contamination is a possibility. When contaminant on one side of the fuel cell passes the membrane and it can contaminate the catalyst on the other side [6]	Comparable effects of all sulphurous species, good filterable with active carbon
NO_x	Strong irreversible or reversible power loss	Adsorption and blocking of catalyst (concurring to oxygen reduction reaction)[8]	Higher impact at high concentration at same total amount will be reduced to NH_4^+
NH_3	Strong irreversible or reversible power loss	$NH4^+$ damages membrane[9][10][11]	Oxidation to NO_2- possible

Table 1 Short overview on air contaminants and pollutants

Roughly 90% of international trade is realised via ships on the sea. Despite the aims of the IMO to avoid air pollutant caused by maritime shipping, most of these vessels are fuelled by heavy fuel oil. This are mainly the residual oils from the refinery processes: it contains significantly more sulphur and other pollutants, for example heavy metals, than fuels used on land. Accordingly, those vessels exhaust pollutes the air quality in port cities and coastal regions especially with sulphur oxides (SO_x) nitrogen oxides (NO_x) as well as soot and fine dust. Even though these pollutants can be strongly diluted on the open sea, they produce harmful effects on fuel cells (and organisms) especially near land and in port. Most ships also sail close to land, where concentrations can accumulate significantly.

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[1] VOC = volatile organic compounds, there are different definitions for this: the European Union defines a VOC as "any organic compound having an initial boiling point less than or equal to 250 °C.

2.3 Identification of operational influences

In principle, the operating conditions for a fuel cell are rather independent of the application on land or on water. Certainly, the individual load profiles differ, but especially in a hybrid concept in combination with batteries, this does not place any special demands or influence on fuel cell operation strategy itself. But, in contrast to applications on land (whether mobile, rail or stationary) and in aviation, there are waves with tilt (rolling and pitching in all three spatial directions), as well as shocks from waves and docking manoeuvres at port facilities. The main operational influences to the fuel cell system will therefore be shock, vibration and inclination.

A) *Shock and Vibration*

A high source of vibration in maritime vessel results from Diesel engines or general internal combustion engines (ICE). Also pumps, propellers, propulsion shafts etc. contribute to vibrations on board. A setup with fuel cell (not producing vibration) and without ICE will significantly decrease vibrations. The specific load requirements of the individual application are widely known and have to be considered in advance by the design of the fuel cell system.

B) *Inclination*

This will result in different inclination of the drive train system and hence the fuel cell. Especially this described maritime operational influences vary of course significantly from concrete application to application and will be very different for all the three Scenarios. So, the impact and effects will also be different.

3. Selection of materials for testing

3.1 Overview of typical components in a hydrogen fuel cell

Fuel cell systems usually consist of a large number of components and parts. Apart from the individual requirements of the different technologies, these are naturally also dependent on the different designs of the individual manufacturers and can vary. Nevertheless, there is a basic arrangement of components that are always present in the different designs. A typical hierarchy of those components, structured in sub systems, subcomponents and used materials (in case of specific for fuel cells) is given in Table 2.

Sub system	**Subcomponent**	**Specialised materials**
PEMFC stack	Membrane Electrode Assembly	
	PEMFC: Membrane	Ionomer Membrane support Additives Other
	SOFC, MCFC, PAFC: Electrolyte	Ceramic
	Catalyst Layer	Ionomer Catalyst material (PGM) Catalyst support Non-PGM catalysts Other
	Gas diffusion layer	Carbon fibre Carbon materials (powders, etc.) Hydrophobic agent / Additives Other
	SOFC, MCFC, PAFC: Porous layers	Metal, coated
	PEMFC: Bipolar plates (BPP)	Coated plate materials Metal based material Carbon based material Other
	SOFC, MCFC, PAFC: Interconnect	Metal, coated
	Seal	Polymers (several, elastomers, silicon etc., metal sheets

	Compression hardware/endplates	
Power electronics / inverters		
System controls	PLC	
Thermal & fluid management	Thermostat Heat exchangers Liquid pumps	
Air management	Air compressor Air filters Air handling / recirculation Air flow meter	
BoP sub components	Filters Valves * Humidifier(s)	
Fuel management	H_2 flow meter H_2 sensor H_2 handling / recirculation	
* Fuel processor / reformer	CO-Clean up Desulphurisation Deionisation	Reformer catalyst Other
* Fuel inlet interface / conditioning	Desulphurisation Pressure control Humidification	
* Heating circuit		

Table 2 Hierarchy of subsystems, components, and materials in a fuel cell (general view)

The greyed-out entries reflect an optional structure, depending on the concrete technology.

To be more specific, based on the P&I scheme in Figure 1, the following Table 3 lists the main components in the respective media paths:

Path	**Component**	**Material**	**Remark**
Air	Air filter	Polymer, nonwoven/ fleece, activated carbon	Replacement part, to be replaced during maintenance
Air	Compressor, Air charger, Blower	Metals (aluminium, steel, copper)	
Air	Tubes	Different silicon grades, hoses with FKM liners, EPDM	
Air	Fittings	Stainless steel, plastic	
Air	Intercooler, charged air cooler	Stainless steel	Optional, depending on compressor
Air	Humidifier	plastic	Optional
Air	Sensors	Stainless steel, plastic	
Air	Sealing materials	Metals, elastomers	
Air	Valves	Metal, plastic	
Cooling	Tubes	Silicon hose	
Cooling	Pumps	Metals (steel, copper)	
Cooling	Mixing valves	Metals (steel, brass)	
Cooling	Expansion tank	Plastic	
Cooling	Fittings	Stainless steel, plastic	
Cooling	Sealing materials	Metals, elastomers	
Cooling	Ion exchanger	Plastic, ion exchange resin,	Optional; replacement part, to be replaced during maintenance
Cooling	Cooling liquid filter	Polymer, nonwoven/ fleece	Optional; replacement part, to be replaced during maintenance
Hydrogen	Water separator	Metal, plastic	
Hydrogen	Valves	Metal, plastic	
Hydrogen	Recirculation	Metals (steel, aluminium, copper) in case of pumps. Metals and/or polymers in case of ejectors.	
Hydrogen	Pipes tubes	Stainless steel, polymers	
Hydrogen	Sensors	Stainless steel, plastic	
Hydrogen	Sealing materials	Metals, elastomers	
Overall	Fuel cell stack	Metal, polymer, graphite	
Overall	Power electronics, Converters	PCB, capacitors, copper	

Overall	(Power) cables	Copper, insulation polymer	Requirement for maritime applications: halogen free insulation
Overall	Other electronic devices	PCB, copper	Must be IP protected
Overall	Control unit(s)	PCB	Must be IP protected
Overall	Supporting frame	Metal	
Overall	Housing(s), Cabinets	Plastics, steel,	Requirement: high IP class for protection of components against water spray

Table 3 Typical main components of a PEMFC system structured by the media

3.2 Components in contact with environmental influences

3.2.1 Chemical environmental influences

Regarding chemical influences, a distinction can be made between corrosion and contamination. In general, it can be said that with a sufficient IP protection class, most components are shielded against the ingress of (salt) water, pollution and the effects of pollutants from the environment. This is why particular importance should be attached to this when designing a maritime fuel cell system. Nevertheless, especially the components of the air system are naturally in direct contact with the outside world. Therefore, the components can be contaminated with pollutants at least during the supply of the reaction oxygen. Likewise, negative influences of impurities in the hydrogen are to be expected, of course, as described above. However, this point can be ensured by QA measures in the origin of the hydrogen. As soon as the hydrogen quality meets the required purity, no negative consequences are to be expected.

3.2.2 Mechanical environmental influences

In principle, a maritime system is also subject to specific requirements regarding shock, vibration, and tilt angles. These are more likely to affect the complete system or larger assemblies such as the stack. There are corresponding load requirement profiles available. Here, too, a significant difference between offshore and inland shipping, with the former having significantly higher requirements has to be mentioned.

3.3 Selection of material and components for operational testing

As described in the previous sections, the entire fuel cell system is naturally considered in the operating methods. If one assumes that no negative effects are to be expected for most of the other components due to the conditions of a maritime application, the actual fuel cell system remains. Therefore, the focus of the corresponding tests is primarily on the stack. In particular, a full stack is planned here in order to be able to investigate the effects of the mechanical influences. In addition, tests are planned to check the influence of salt spray on fuel cell operation. An exemplary setup in the form of a short stack is sufficient for this.

3.4 Selection of material and components for ex-situ testing

Tests are planned to check the influence of salt spray on fuel cell operation. An exemplary setup in the form of a short stack is sufficient for this. For some investigations, test methods are available in which the environmental effects do not have to be examined on a complete system, but rather the relevant assembly or an individual component in an ex-situ test. Based on the assumptions described and the components from Table 3, the following selection of components was chosen for these tests (see Table 4):

Component	**Test procedure**	**Partner**
Cathode hose material	200-500 hours durability study using 8-cell 25 cm^2 MultiSingleCell setup with individual gas feeding. Thermal decomposition study including gas analysis to try to identify contaminant species.	VTT
Anode hose material	200-500 hours durability study using 8-cell 25 cm^2 MultiSingleCell setup with individual gas feeding. Thermal decomposition study including gas analysis to try to identify contaminant species.	VTT
Plastic valve and/or ejector materials for cathode	200-500 hours durability and CO tolerance study using 8-cell 25 cm^2 MultiSingleCell setup with individual gas feeding.	VTT

Table 4 Selection of components for Ex-situ Testing

e-SHyIPS is a project founded by the European Union Framework Programme for Research and Innovation Horizon 2020 under GA n. 101007226

4. Test setup VTT

4.1 MultiSingleCell for contaminant measurements

For contaminant research the screener stack (also known as MultiSingleCell, MSC) concept developed by VTT, will be applied and further developed [14]-[17]. The screener stack concept and cell versions 3 cm^2, 10 cm^2 and 25 cm^2 were developed by VTT in years 2006-2010 and applied in fuel contaminant, membrane durability, GDL durability, BP plate corrosion and catalyst screening work. A schematic diagram of one MSC type with its flow arrangement is shown in Figure 3. Proton Motor will therefore send MEA components (CCM and GDL) to VTT on demand. Multi-cell test stands have also been developed by MEA developers, such as 3M [17]. 3M had a test stand which could operate ten 50 cm^2 single fuel cells simultaneously at different load settings and cell temperatures using a single gas stream and humidification system. One of the key ideas of the test rig at VTT is the flow splitting using precision orifices (additional flow resistors, Figure 3 b) as it enables very accurate and cost-efficient control of the flows. Individual control of cell current is also possible if multiple electronic loads are used. All the cell inlets and outlets can be separated and cooled so that condensed effluent water can be analysed e.g. for contaminant emissions from dried gas flows. Up to eight cells can be run at the same time with a single fuel cell test station. Typically, when materials are tested, two reference cells (no contamination) are used as well as 3 x 2 sample cells or 2 x 3 sample cells. The screener stack arrangement will be used in the e-SHyIPS project as follows. Up to three different materials (e.g. different hose or valve materials) will be run in parallel under the same conditions having the same experimental history. Accelerated conditions can be created by keeping the studied materials at maximum expected operating temperature.

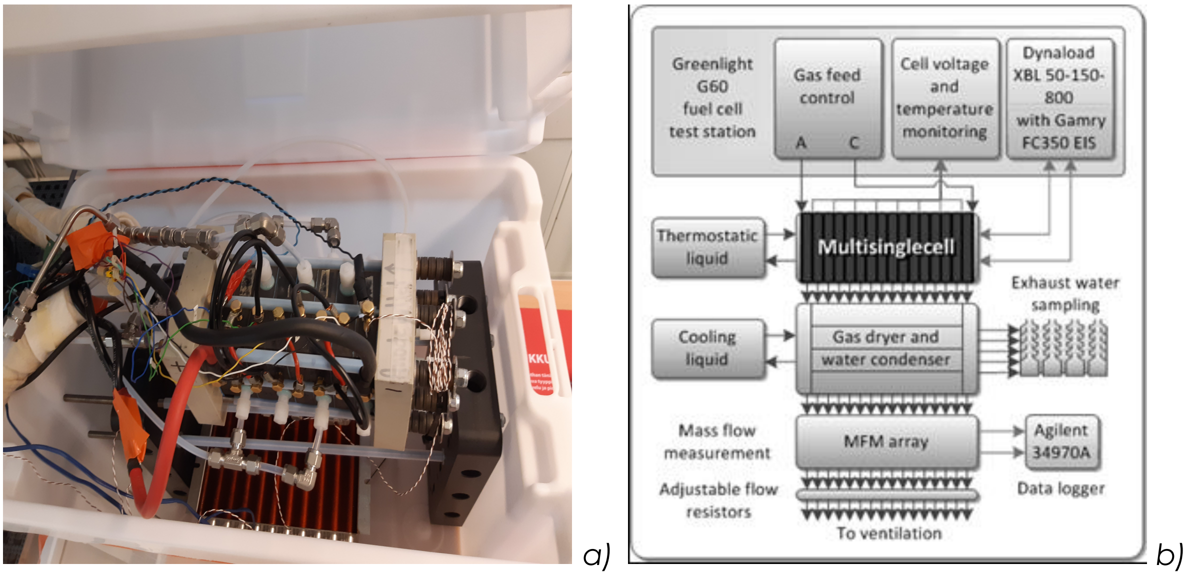

Figure 3 *a)* screener stack (a 25 cm² version with 4 cells) at VTT; 3 *b)* the flow arrangement of a MultiSingleCell at VTT with controllable and measurable exit flows.

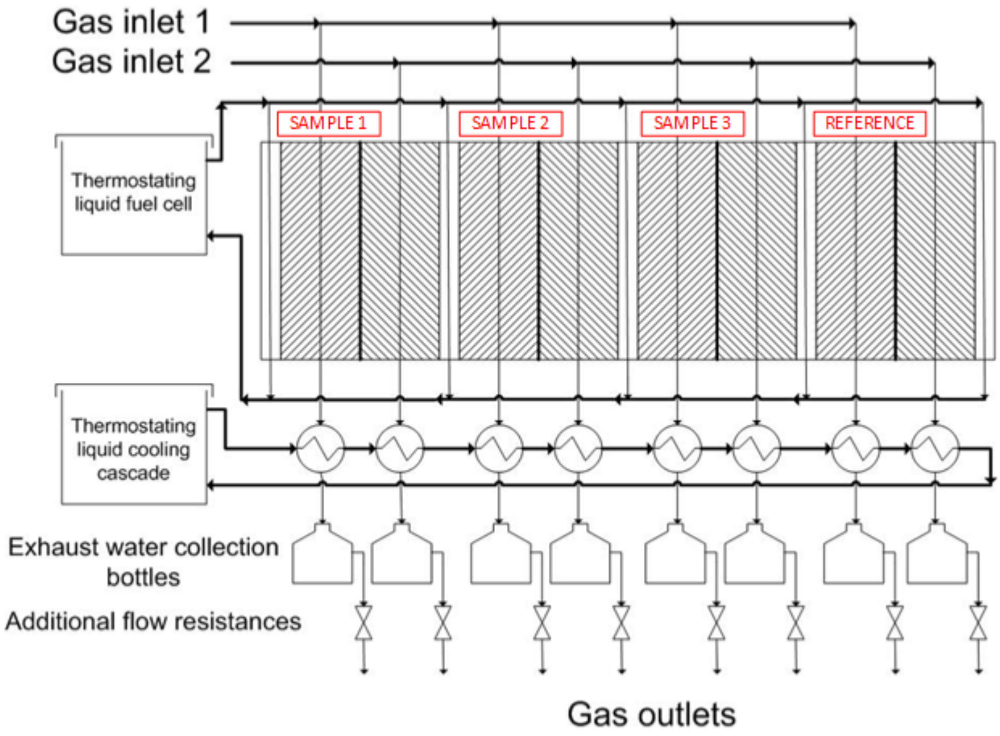

Figure 4 Schematic of the MSC gas and thermostatic fluid manifolds. The studied sample materials are placed before cells and heated to the desired temperature.

4.1.1 MEA break-in and recovery protocols

MEA break-in has significant influence on cell performance during operation. Several protocols are available from the scientific and patent literature. Common elements in all break-in procedures are the oxidation of additives and degradation products used during MEA-manufacturing, reduction of Pt surface oxides, and the reductive desorption and subsequent washout of additives and degradation products from MEA-manufacturing.

Since MEA break-in and recovery protocols are dependent on MEA manufacturing process, these will be selected based on recommendations from MEA suppliers. If recommendations are not available from MEA manufacturers, then cathode starvation with high humidity is used, as proposed in literature.[18]

4.1.2 The measurement procedure

For performing the screener stack measurements, the procedures (test modules and test programs) and recommendations from the FCH JU Stack-Test project (Grant Agreement No. 303445)[19] will be reviewed and adapted for the needs of the project. The measurement is shown Figure 5 and is described as follows:

1. A sufficiently long MEA break-in period and period without material samples is needed to have a clear value for steady-state reversible degradation.
2. The gas flows are lead through the studied materials and materials are heated in steps (e.g. every 24/48 hours) from 60 °C to maximum expected operating temperature.
3. When the samples are heated to the maximum expected operating temperature, the mitigating effect of start-up and shut-down is studied by performing maritime application relevant start-up and shut-down procedures.
4. The reversibility of contamination is studied when gas flows are by-passed. First, 24/48 h steady-state operation is applied and then application relevant start-up and shut-down procedures. Finally, the cell temperatures are lowered to study the "wash-out" effect.[20]

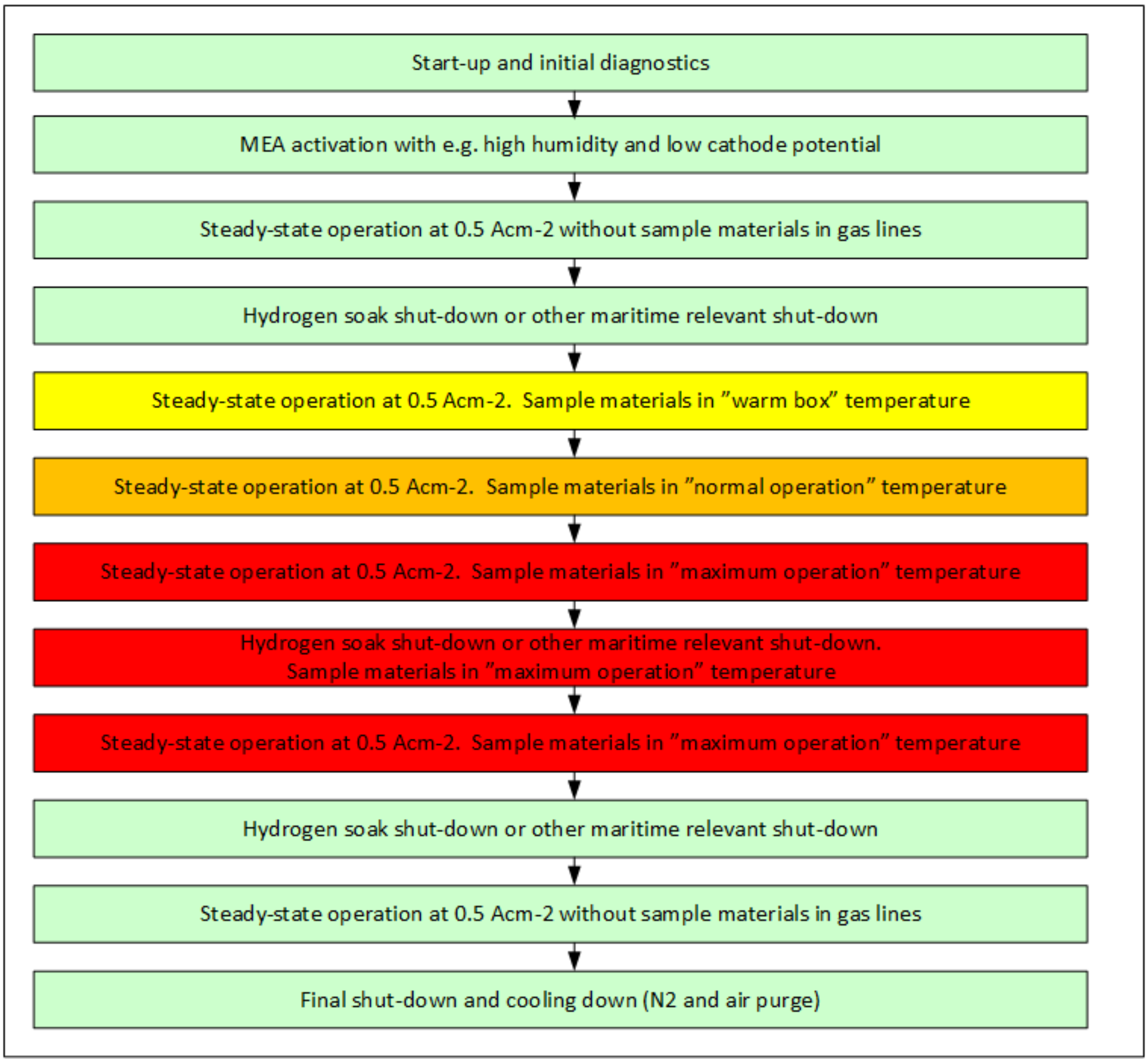

Figure 5 The planned measurement procedure.

When the measurements are done for materials to be used on the cathode or on the anode side, the measurement procedure is adapted according to expected temperatures and reactant utilisations.

Measurements for the material used on the cathode side

- Maximum temperature up to 110 °C or even more if materials after compressor and before humidifier are studied.
- Relatively high stoichiometry is used in the measurement to reach stable performance.

4.1.4 Measurements on the anode side

- First screening measurements are done using open anode configuration with relatively high stoichiometry.

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- In addition to performance, also CO tolerance will be studied using methodology developed in EU funded HYDRAITE project (Grant agreement ID: 779475) [21].
- The most promising materials, which have almost zero effect, are tested also with high fuel utilisation (>98%) using hydrogen recirculation.

4.1.5 Selected operating conditions

The selected operating conditions correspond maritime application. However, the maximum operating temperature of MSC in "warm box" is about 70-75 °C. Dew points up to about 65 °C can be used. CO desorption or H_2 adsorption are potential in-situ characterisation methods, which can be applied before and after the measurements. However, the use of cyclic voltammetry may also clean the catalyst surfaces from contaminants as exposure to high potentials can oxidise some of the contaminants [22].

4.2 Evaluation of test results

The results are evaluated so that irreversible degradation is separated from reversible degradation. The methodology developed in STACK-TEST [19] project for air and fuel contaminants will be adapted and used. Contamination is monitored by comparing steady state performance of cells when they are run at about 0.5 Acm^{-2} constant current as well as measuring polarisation curves periodically. For anode side, the contamination is also monitored by measuring remaining CO tolerance of the anode compared to new electrode. This is done by measuring the time it takes to have a small voltage drop (30-50 mV), when CO containing fuel is used. The expected CO level is 2-5 ppm.

5. Test setup at PROTON MOTOR

As Proton Motor is equipped with all necessary and suitable test benches in their own facility, the consortium decided to carry out operational tests at the Proton Motor short stack test benches. For the planned tests, both Proton Motor and VTT, as the scientific advisor, found a test on short stacks to be perfectly adequate. A so-called short stack essentially consists of the same components as a full stack, but with a significantly smaller number of cells. This ensures that the effort and the associated costs are significantly reduced. This is especially applicable in the case of a damaging test as planned within this WP4. To simulate the real behaviour of a full stack, an ensemble of at least five cells is necessary in order to avoid edge cell phenomena, for example due to cooling. For this reason, short stack tests are also preferred to single cell tests. The commercially used stacks at Proton Motor consist of 96 to 144 cells, although in case of metallic bipolar plates the stack may consist of a significantly higher number of cells, in the range of 300 to 400.

Figure 6 Proton Motor FCS test lab with short stack and full stack test benches

Three proprietary short-stack test benches are available at the Proton engine plant in Puchheim, Germany as shown in Figure 6. Due to their special design, these can be adapted to the corresponding requirements of the test plan for extensive test procedures. In addition, non-destructive tests are also planned, which can be better reproduced on a full stack. Proton Motor also has several test stands available for this purpose. The concrete test plan is described in the following section. The above Figure

6 shows the Proton Motor test field with the short stack test benches (foreground) and the full stack test benches (background).

5.1 Test plan

Stack tests at Proton Motor will be differentiated according to the size of the stack to be tested. PM plans to conduct test series on a short stack level, as described above, consisting of five cells. Additionally full stack tests will be performed with the stack consisting of 96 or 120 cells. All tests, regardless of the stack size, will be performed with preconditioned stacks (following standard PM procedures) in order to guarantee comparable and full performance at the beginning of the test series. At the short stack level experiments for salt spray contamination as well as operating strategies will be tested. Due to the high sensitivity of the catalyst towards contamination and different operating strategies, it is sufficient to perform these experiments on the short stack level. Moreover, the salt spray tests will critically and irreversibly damage the stack and the material investment (including H_2 for operation) would be too high for a full stack.

5.1.1 Characterisation methods

Evaluation of the contamination effects can be done by holding the stack at a constant operating point (current controlled) and observing the change in cell potential after the injection. Furthermore voltage-current (U/I) characteristic curves are recorded before and after contamination, which give indication to the stack performance over the full operating range (power output). This U/I characteristic curve (also called *polarisation curve*) as exemplarily shown in Figure 7 is a common method to determine the performance characteristics of a fuel cell stack, regardless of the fuel cell technology. Even different types of fuel cells can be compared. But more importantly in this case, degradation effects (reversible or irreversible) can be easily detected and furthermore, cell level effects (i.e. location of damage and inference of mechanism) can be determined. An exemplary curve is shown in the following Figure 7:

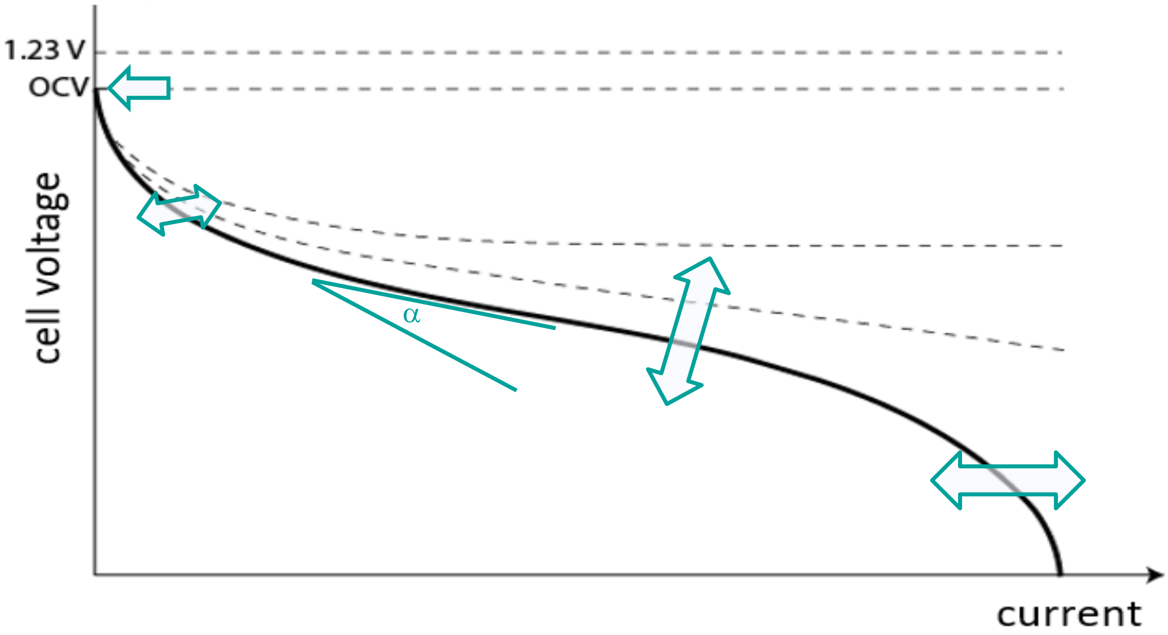

Figure 7 Exemplary U/I characteristic or polarisation curve of a fuel cell

If no current is drawn, the so-called *open-circuit voltage* (OCV) is established. Due to various losses, this is significantly below the theoretical normal potential of the chemical reaction at about 1 volt. As soon as current is drawn from the system by an external consumer, the voltage drops along this curve. In case of damage due to various influences, the value of the OCV, the slope of the curve, the position of the bend in both the front and the rear area and the general position of the linear area can be shifted. This allows conclusions to be drawn about the type and species of poisoning or harmful influence and measures for prevention to be defined accordingly.

5.1.2 Salt spray contamination tests

For these experiments a salt solution has to be prepared, using de-ionised (DI) water. For simplicity reasons only sodium chloride (NaCl) will be used. Concentration will be adjusted to represent different environmental conditions for the ship operation and might also be varied throughout the test series. Injection of the salt spray into the fuel cell will be conducted using a nebulizer and the amount and/or interval of injection will also be varied. Additionally, the product water (air side) will be analysed for rest salt content. This will allow conclusions about how much salt is retained in the stack and the cells. Post Mortem analysis of the cells will also be done at VTT. Via microscopic techniques (e.g. SEM and EDX) it can be ascertained where the salt and the corresponding ions are accumulated and whether certain damages can be seen, for example punctures in the membrane or blockage of flow field channels.

5.1.3 Operating points

In order to test behaviour of the stack at different operating points, mainly load levels (current controlled) will be varied, which will generate different power outputs. Beside the load level, the load dynamics are a crucial characteristic of the fuel cell, which will immensely influence the lifetime. This can be tested by varying the power output over time; in terms of the current ramp (A/s) during the change, as well as the number of load changes or cycles per time unit. Within the tests of the dynamics complete shutdown/start-up cycles can be performed as well. Generally, a higher dynamic will lead to increased degradation and the degradation in µV/h at a certain load is one way of analysing the data following the test series. Also, as above, UI-curves are recorded at regular intervals to compare performance over lifetime.

5.1.4 Inclination behaviour on full stack level

Concerning the inclination, the most crucial aspect is the removal of the product water that is generated in the electrochemical reaction. As the amount of produced water is much larger in full stacks, it does not make sense to perform these measurements in a short stack setup. Ideally the stack is oriented in such a way that the media outlets (especially air and hydrogen) are facing downwards so that gravity assists the removal of water. In case the stack is integrated into the system in a standing position, i.e. all cells are oriented parallel to the ground and all media outlets are facing downwards, the water removal should not be an issue, even if inclination in all axes with angles (α) of up to 45° is taken into account. However, sometimes it might not be possible to orient the stack in such a way, e.g. due to installation space restrictions. In order to test the influence of the stack inclination in case of a lying stack (see Figure 8) it will be operated at constant operating points and the inclination will be varied. If possible, the inclination in different axes will be varied and evaluated, although the tilting towards the back with the media outlets facing upwards is the most critical position as water removal is the hardest task. The background for this test is: in addition to the damage caused by contaminants described above, there is also damage caused by the liquid product water generated during the chemical reaction. During flooding, this can block the catalyst that is important for the reaction and, in addition to an acute loss of conductivity, also contributes to instantaneous and/or continuous ageing. Through a suitable configuration of the stack and its spatial location as well as a corresponding operating strategy, care must be taken to ensure that such flooding cannot occur. The aim of the proposed tests is to explore the limits of possible operation with regard to different spatial orientations (3-dimensional) of the stack.

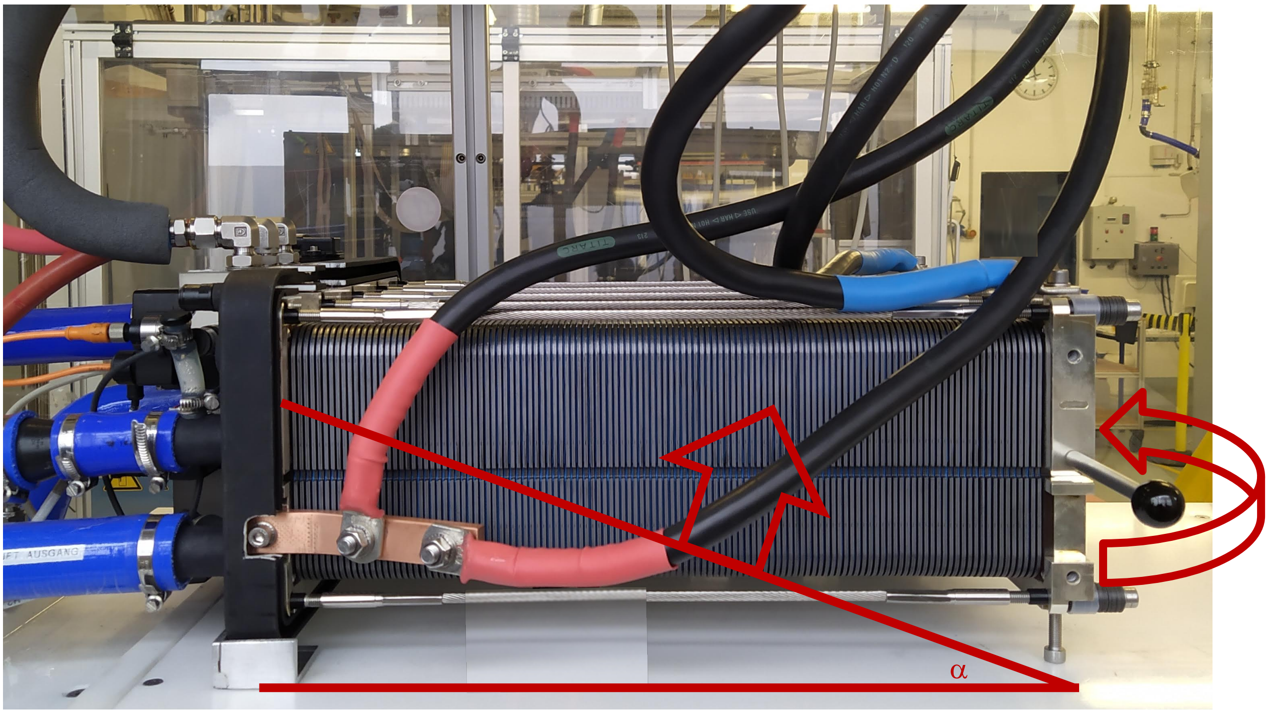

Figure 8 Full stack at the PM test bench - visualisation of inclination angles

During the constant operation it can be ascertained at which inclination angle the water removal becomes so difficult that the catalyst is flooding and no stable operation will be possible anymore. This can be seen at the cell potential dropping down and becoming more purge sensitive. Additionally the operating points can be adjusted to return to a stable performance at inclination, e.g. with a higher air flow rate or higher stack temperature.

5.2 Test bench adaption

With regard to the tests to be carried out and the test plan to be developed, some adjustments to the laboratory test benches must be made.

Concerning the short stack test bench mainly the air supply is affected. For evaluating the effect of salty air on the fuel cell the introduction of a nebulizer into the air supply is necessary. Particular care must be taken here to ensure that the test bench is not damaged by the negative effects of the salt air (e.g. through corrosion or poisoning through crystallisation in the pipes): the complete air supply piping starting at the nebulizer until after the stack has to be adjusted so that it can be exchanged after the tests to not permanently contaminate the test bench. This includes possibly some sensors that are integrated into the test bench, such as pressure and temperature sensors in the air supply.

Additionally, the product water has to be condensed and separated after the stack to analyse for salt content. Considering the harmful effect of salt spray and the damage to the stack, it is advised that such experiments will not be carried out on full stack level. However, these tests on stack orientation only make sense in a full stack configuration, as the expected effects may not be as distinct in an exemplary short stack configuration. Therefore it is planned to test stack inclination on the full stack test bench, in order to simulate stack position on a highly turbulent sea. Therefore the stack has to be mounted on a support that will allow it to be tilted. In the test benches at PM the stack is mounted in a lying position, i.e. with the active area of the cells orthogonal to the horizontal plane and the media connections on the front; compare also Figure 8. If it is not possible to find or implement a mount that will allow inclination in every direction, only the inclination towards the back of the stack will be realized and tested. In any case this is the worst case for the stack operation, as the media connection will be facing up, which negatively affects product water removal. Moreover, in order to allow tilting of the stack the media piping has to be made more flexible.

6. Post mortem analysis

Post mortem analysis of materials and components will be carried out at VTT in close collaboration between WP4 partners. Selected materials and components from both PM and VTT experimental testing will be analysed at VTT using relevant analysis methods at hand, such as SEM and EDX. For this purpose, VTT has reading laboratories that are equipped with a wide variety of examination and analysis methods in order to be able to handle the respective analysis task at a high scientific level according to the specific requirements. Some of these specific test requirements and questions can only be determined in the course of the test phase. Therefore a more detailed post-mortem analysis plan will be finalized after getting the first results from the experimental test campaigns.

6.1 Time schedule

The tasks described in this document have to be aligned with the overall project's progress. However, careful scheduling is important because some of the experiments are time-consuming tests with open results. Therefore, sufficient buffer time must be planned in order to be able to react to unforeseen effects or influences. The initial time planning can be seen in Table 5.

Task	**Partner**	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
Adapting test benches	PM	x	x	x	x	x									x	x	x									
Salt spray contamination tests	PM					x	x	x	x	x	x	x														
Operating points	PM									x	x	x	x	x	x	x	x									
Inclination behaviour	PM															x	x	x	x	x	x	x	x	x	x	
Ex-situ tests	VTT				x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x		
Post mortem analysis	VTT											x	x				x	x						x	x	
Deliverables	VTT													x												x

Table 5 Time schedule for testing

7. Conclusion

In scope of the work of this deliverable typical components of a fuel cell in the maritime sector were worked out, as well as the main factors of the environmental conditions that lead to negative or critical effects. Subsequently, materials, and components were selected and appropriate test procedures were defined by which the discussed stressors can be examined more closely so that their harmful influences on the fuel cell system can be circumvented. The resulting test plan serves as the starting point for the following work of WP4, namely the actual tests and test procedures. Due to the fact that findings are obtained during the experimental tests, which may influence the test procedures, it is expressly stated here that the actual procedures may change in terms of both time and scope in order to adapt to any new potential test results. Results will be presented in the following Deliverables D4.2 – *Interim test results from material and component testing and post-mortem Analysis* which is due in M24 and the final results in D4.3 – *Test results from material and component testing and post-mortem Analysis*, due in M39 as conclusion of the activities T4.2/T4.3/T4.4

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