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Kansen voor West 2 – Field lab electrification (FLIE)

Deliverable: WP3 Power to Chemicals

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1 General Introduction

The regional working group Klimaatakkoord industrial cluster Rotterdam-Moerdijk identifies in its report “In three steps towards a sustainable Rotterdam-Moerdijk industrial cluster in 2050” electrification and green hydrogen as important solutions for the transition to a new energy system in 2030 and a new raw materials and fuels system in 2050. These developments concern deployment of renewable energy in industry, also called industrial electrification, or Power-2-X.

Ambitious goals regarding the realization of wind farms in the North Sea and the Canal area will greatly increase the variability of electricity supply in North-West Europe. Converting electricity into fuels and chemicals can convert large amounts of green electricity into energy-rich green chemicals suitable for long-term storage and for use in the chemical industry.

Power-to-X (with different possible chemicals for X) is therefore both a solution for the energy system and a solution for the chemical industry.

This industry is under increasing pressure to reduce CO₂ emissions while there are limited options to achieve the substantial emissions reductions required. The use of renewable energy to replace fossil fuels and at the same time use CO₂ as a raw material is therefore an attractive option.

A number of Power-to-X technologies that can convert CO₂ into a circular chemical product have been demonstrated in the lab.

However, these new technologies are not yet easily adopted by the industry. The risks are great and new solutions are not yet affordable. There are many technical, operational and market risks that play an important role in acceptance and implementation of electrification technology. Collaboration and chain thinking are therefore necessary.

Because of the above challenges, needs and analysis, the regional and national partners Deltalinqs, FME, InnovationQuarter, TNO, Port of Rotterdam and the Municipality Rotterdam, in consultation with the public and private partners of the Rotterdam Climate Table, together took the initiative with a number of SME frontrunners to launch the Industrial Electrification Field Lab (FLIE)

By bringing together existing networks, knowledge and infrastructure, public and private forces are joined and players from the process industry and innovative SMEs can be helped more effectively and faster.

Within the FLIE framework, the Kansen voor West 2 project ‘FLIE’ was developed. In this project, a number of Low TRL Power-to-X technologies are developed to higher technological maturity, up to a level that the technology becomes sufficiently mature to move it out of the lab environment and into an industrial environment in the Field lab, where it can be integrated into the full product chain and becomes available to FLIE partners and clients for further experiments, industrial environment testing and demonstration.

This document is the report on Workpackage 3 in the project, ‘Initiating and executing Power-to-Chemicals projects’.

The port of Rotterdam is a very promising location for Power-2-Chemicals projects because i) the expected availability of green electrons, hydrogen, CO₂ and residual heat and the infrastructure for these raw materials

ii) synthetic fuels and chemicals are very strategic for the future of the established petrochemical industry

iii) the industrial environment with potential operation partners and chemicals off-takers offers a very relevant experimental space for SMEs.

The goal of this Workpackage was therefore to initiate, develop and execute two research projects on Power-to-chemicals.

The chemicals selected in this project were dimethylether (DME) and formic acid (FA).

Power-to-DME project

DiMethylEther (DME) is a promising synthetic replacement for diesel and can be used in heavy transport, especially in shipping and trucks, reducing CO₂ and particulate matter emissions. In addition, DME as a chemical is an important building block for the synthesis of aromatics and polyolefins in the chemical industry, analogous to methanol, for example. In the Power-2-DME concept, green hydrogen is first produced using Proton Exchange Membrane Water Electrolysis (PEM-WE) technology, after which the CO₂ with this green hydrogen is converted into DME using Sorption Enhanced DME Synthesis (SEDMES) technology. These technologies have been developed individually up to small-scale pilots in previous projects, but now need to be further optimized and combined. The current project focuses on further developing the small-scale pilots developed in previous projects and subsequently connecting them together and testing them in the integrated situation, preparing for transfer to an industrial environment. SME company Hydron Energy/ Schaeffler (electrolysis) has an interest in this technology being further developed and demonstrated more quickly in a relevant industrial environment, so that it can then be scaled up. The work done in the Power-to-DME project is reported in Chapter 3.

Power-to-FA project

Formic acid (FA) is the chemical that can be produced most efficiently from CO₂ and electricity. This is done through a process of direct electrochemical conversion. Formic acid is a well-known product with its own existing but relatively limited global market. Formate is the salt of formic acid, and also has its own market. Producing formate and/or formic acid in a sustainable way creates several possibilities to increase the use of formic acid:

Green formic acid can replace existing 'grey' formic acid to help companies reduce their carbon footprint;

Green formic acid can also be used for processes where acids are required for conditioning processes, such as water purification; here, for example, hydrochloric acid and/or sulfuric acid can be displaced;

Formic acid can also be used as a nutrient source for fermentation processes, which aim to produce, for example, ethanol, fatty acids or bioplastics; sugars can be replaced, thereby reducing competition with food production;

In addition, formic acid is an excellent carrier of hydrogen and carbon monoxide, and thus functions as a liquid syngas that can be easily stored and transported; formic acid can therefore also be used for energy storage.

TNO and COVAL have been working together for a long time to develop and scale up the electrolysis process for the production of formic acid.

In the Power-2-FA project, CO₂ is converted directly into formic acid in a high-pressure electrochemical reactor. This reactor was developed by Coval Energy into a working concept

in the TU Delft laboratory in previous and ongoing projects (RVO projects P2FA and Phecarn), but must now be further developed into a pilot installation in an industrial environment.

The proposed project focuses on the optimization of formic acid production in a reactor with gas-diffusion electrodes (GDE's) and in stacked electrochemical cells. Sufficient information must be obtained through testing in a relevant industrial environment to develop a detailed design of a pilot installation that can be used in an industrial environment in a follow-up project.

The SME company involved, Coval Energy, has a great interest in developing this technology as quickly as possible towards a pilot installation, and then a demo installation, to enable the reuse of CO₂ via this route on a commercial basis.

Testing of the electrolyzer stack developed by COVAL is made possible by the design and construction of a pilot scale electrolysis installation called ZEUS, developed by TNO.

The proposed combination of technologies and long- and short-term R&D, combined with serious industry involvement is unique and will lead to much faster development and demonstration of the technologies and their potential for partial decarbonization of both industry and transport. The availability of the CO₂ electrolysis installation in the Field lab after the end of the project is a unique feature and will attract future interest from research and industry.

The work done in the Power-to-DME project is reported in Chapter 4.

2 Power2DME

2.1 Introduction to P2DME

The considered power-to-chemicals process to produce Dimethyl ether (DME) from CO₂ and H₂ from a water electrolyser (WE) running on renewable energy, is depicted in Figure 1. It consists of three main parts: i) a Polymer Exchange Membrane (PEM) electrolyser incl. H₂ pressurisation, ii) the SEDMES unit and iii) distillation train for DME purification. The different parts were or are being realised within previous projects, RVO NextgenH2, INTERRED E2C and EZK FLIE lab, respectively. The overall P2DME process has been evaluated in terms of economic feasibilityⁱ and carbon footprint. In this project the actual connection between the PEM electrolyser and SEDMES pilot will be designed, engineered and built. This will lead to the first experimental proof of concept of the P2DME technology. Furthermore, as the H₂ is the main cost driver for the P2DME process, improvements in the PEM electrolyser stack design are key to make the process economically feasible. A low stack performance degradation, high energy efficiency while reducing cost, are the prime drivers behind current PEMWE stack development and design.

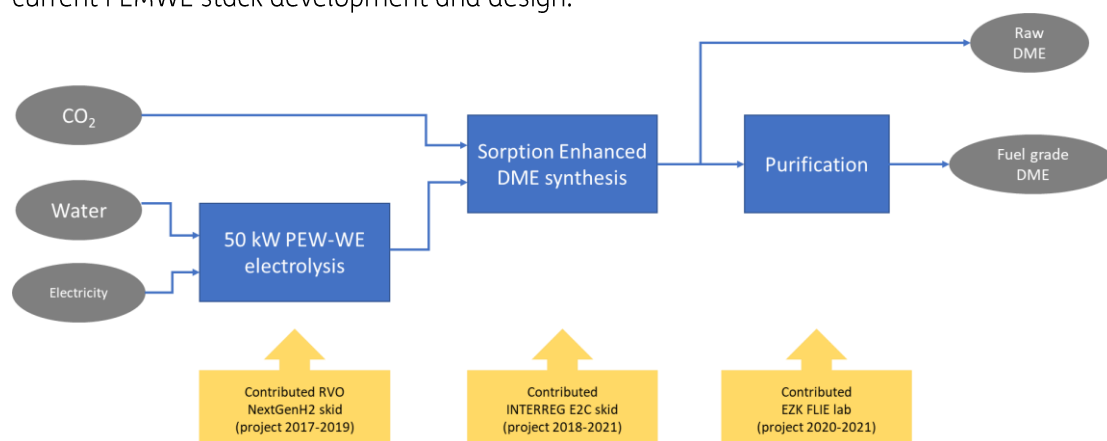


Figure 1: Basic structure of inlet and outlet streams of the Indirect Route for the production of DME from CO₂, electricity and water.

DME is a friendly commodity chemical (4 megaton annually) and finds its way to consumers as a propellant in aerosol products, such as hair spray. Renewable DME (rDME) is a high potential candidate to replace diesel in heavy duty machines where it has the major advantage that it combusts without soot formation. In addition, rDME is one of the primary candidates to replace LPG and serve as the fuel of choice in difficult to decarbonize (off-grid) sectors and as such eliminating the dependency of these sectors on petroleum-based fuels. Last but not least, DME is seen as a key building block in a future circular carbon economy.

Two methods can currently be employed for DME production: the indirect method and the more recent direct synthesis approach. In the conventional indirect method, methanol is first produced and then dehydrated to create DME, involving two separate reactors with thermodynamic limitations and the need for extensive separation methods. In the direct synthesis, both methanol synthesis and dehydration take place in a single reactor with a bifunctional catalyst, resulting in higher single pass conversion and a higher DME yield. Challenges include maintaining reactant balance and preventing carbon atom loss.ⁱⁱ

SEDMES is a novel process which provides another conversion route from syngas to DME, which is being developed at TNO. SEDMES is a reactive adsorption process, in which water is removed in-situ by means of a solid adsorbent. By the removal of H₂O as a product, the oxygen surplus of the syngas feed no longer ends up in CO₂. The single pass conversion is SEDMES is higher than in the direct or indirect DME synthesis route. Consequently, the recycle streams in SEDMES are smaller. Other advantages include enhanced catalyst lifetime and boosted process efficiency, specifically in the case of diluted CO₂-rich gas streams. Lastly, it enables the use of CO₂ as a feedstock.

2.1.1 Sorption enhanced DME synthesis

Separation enhanced synthesis routes have shown to offer major advantages in the conversion of carbon dioxide to chemicals including DMEⁱⁱⁱ. In fact, SEDMES has proven to yield a very high carbon selectivity to DME. SEDMES is a reactive process, in which steam is removed in situ by using a sorbent. As a typical pressure-swing-adsorption (PSA) system, it undergoes a cycle of reactive adsorption and sorbent regeneration, as illustrated in Figure 1. A typical SEDMES cycle consists of at least the following four consecutive steps:

- i) Adsorption (ADS) – the feed is converted into DME on the catalyst, while steam is removed by a zeolite adsorbent;
- ii) Blowdown (BD) – counter-current depressurisation of the system in order to desorb water;
- iii) Purge (PURGE) – counter-current flow of dry hydrogen to remove water from the column;
- iv) (REP) repressurisation – co-current pressurisation of the column with the feed stream.

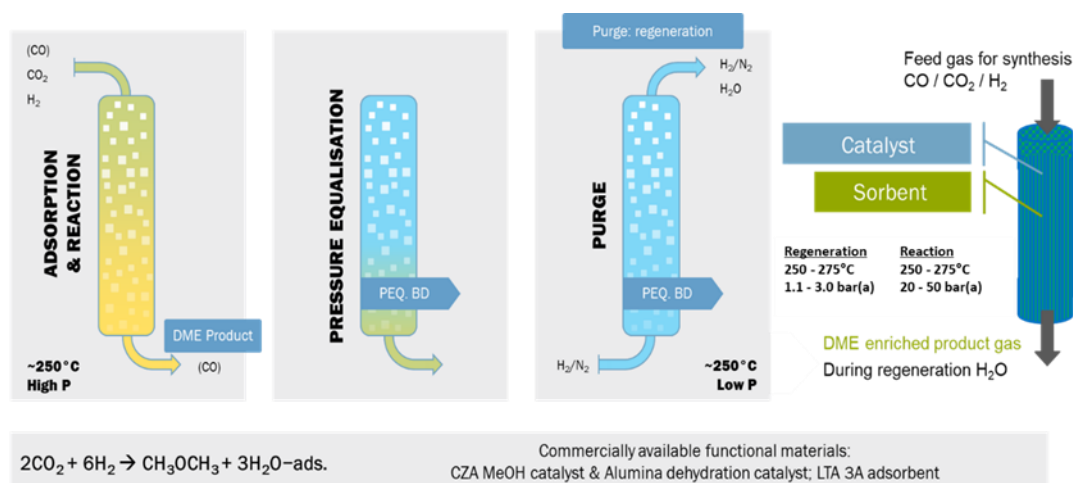


Figure 2: SEDMES cycle with the consecutive steps adsorption (ADS); Blowdown (BD); PURGE and repressurisation (REP).

The SEDMES technology has been successfully tested on lab and bench scale, making use of existing infrastructure of other sorption enhanced processes. In the Interreg E2C project the first dedicated SEDMES facility has been built for scale-up and further development of the technology.

2.1.2 PEM development

One of the main cost drivers behind the levelized cost of hydrogen (LCOH₂) produced by electrolysis is the cost of electricity and consequently the energy consumption to produce 1 kg of hydrogen. Apart from the efficiency, that can be expressed as Kg/kWh, stack durability and degradation also play a major part in the LCOH₂. To optimize and increase the overall stack performance, Schaeffler's development focus is on improving the catalyst coated membranes in order to increase performance, and the application of novel stack components and coatings to increase lifespan while decreasing degradation and cost.

2.1.3 Objectives of the P2DME technology line within the KvW2 project

The main goal of the P2DME technology line within this project is to demonstrate the process experimentally combining the 50 kW PEM electrolyser and SEDMES unit (Figure 3) in FLIE Petten.



Figure 3: Nextgen container (left bottom container), and SEDMES unit in FLIE Petten.

The following objectives were set for the P2DME development within the KvW2 project:

1. Improved PEM-WE pilot installation with enhanced materials, Design for Manufacture and Assembly (DfMA), and upgraded Balance of Plant for hydrogen conditioning.
2. Enhanced SEDMES pilot installation with improved flexibility, stability, and increased pressure to optimize productivity.
3. Connection of PEM-WE and SEDMES installations with skid interconnect design and relocation of SEDMES to the field lab.
4. Commissioning of the downstream purification unit (DPU) and skid interconnect for SEDMES and DPU columns.

5. Test program results for DME production, an accredited test program report with findings, conclusions, and recommendations for further development steps.

This chapter describes the results from the project on the P2DME line.

2.2 Technical report

2.2.1 NextGen Electrolyser Upgrade

Before the start of the KvW2 project the Nextgen WE was out of operation for several years. It was developed within the NextgenH2 project from 2017-2019 and was operated for several weeks then before the testing was stopped due to issues with the cooling system and H₂ pressure control.

The NextGen system has been upgraded with the following equipment to provide stable and reliable conditions for the PEM stack operation (Figure 4):

1. New extended cathode gas/liquid separator for better control over water draining and separation
2. Improved stack process connections
3. Conductivity monitoring of cooling loop and anode loop
4. Low conductivity propylene glycol as the cooling liquid inside of the cooling loop
5. Cooling loop copper air-cooler exchanged for a stainless steel one to maintain low conductivity of the cooling liquid.
6. Improved water purification of anode process water
7. Flash vessel for a safe release of dissolved hydrogen
8. Inline binary gas analyzer for detection of hydrogen in oxygen concentration.



Figure 4: Pictures of main equipment for improved PEM testing station. The numbers correspond with the list above.

2.2.2 Stack development & testing (Schaeffler)

During the initial phase of the project Schaeffler provided engineering support in improving the system as described above. Various technical discussions were held on-site and remotely. Furthermore, Schaeffler defined a detailed test plan and protocol to successfully test and operate its EL500 PEMWE stack platform.

In the pursuit of improving the performance and durability of the Hydron K-100 platform, rated at a nominal capacity of 100kW (2,2 KgH₂/hr), Schaeffler build over 20 single cell stacks with different materials and configurations and tested these on key factory approval indices.

Furthermore, Schaeffler provided a 30 cell EL500 commissioning stack for system testing and commissioning purposes (Figure 5). This commissioning stack was operated for more than a 1000 hrs in the KvW2 system and served as an important tool for verifying and improving the system performance and reliability. A stable balance of plant is key in obtaining good quality PEMWE performance and degradation data.



Figure 5: EL500-30 cell commissioning stack in PEM testing system

Apart from the commissioning stack, a second EL500 stack was built with next generation CCM technology, that will also be deployed in the 100kW K100 platform. This stack has been also qualified and characterized by an external party, enabling Schaeffler to learn more on the stack, comprising novel materials and CCM, performance and operating behaviour in different test systems and environments.

Lastly, Schaeffler installed its remote monitoring box in the KvW2 PEMWE pilot system to have direct access to the data and to test its cloud-based data handling and storage system.

2.2.3 P2DME: integration electrolyser SEDMES

During the project the FLIE lab location at Plant One Rotterdam was discontinued and therefore all activities including the integration with the WE were performed in Petten. For the integration of the Nextgen WE and the SEDMES, the outlet of the cathode of the Nextgen had to be connected to the hydrogen process inlet of the SEDMES (Fout! Verwijzingsbron niet gevonden.6), taking the considerations below into account. The P&ID of the connection is depicted in Appendix 4.1.

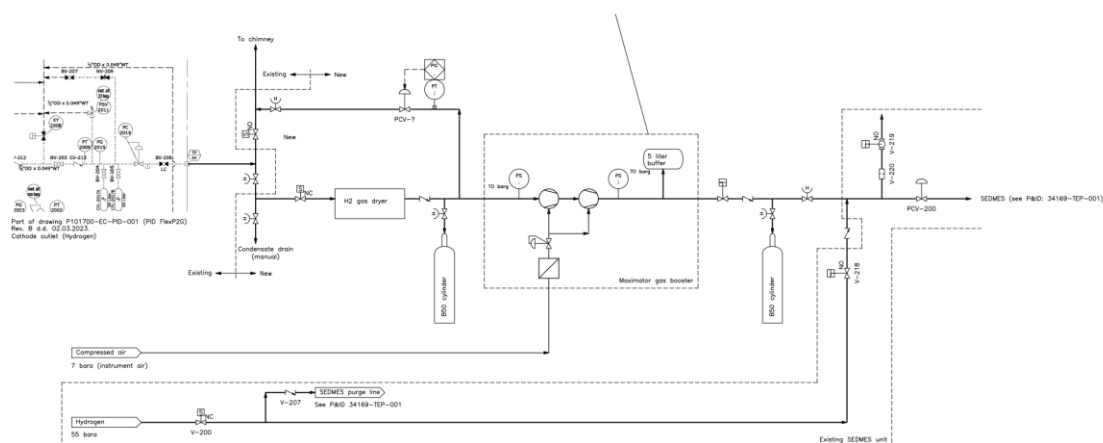


Figure 6: PFD connection of the Nextgen and the SEDMES units

Pressure: The maximum operating pressure of the Balance of Plant (BoP) of the Nextgen was 25 bar, but the preferred operating pressure of the SEDMES unit is 30 barg or higher. Therefore, extra compression of the hydrogen was required.

Humidity: The humidity of the hydrogen is important to prevent damage to the hydrogen compressor and downstream in the SEDMES process. Thus, the hydrogen needs to be dried before compressed.

Flow: The maximum hydrogen production flow of the Nextgen depends on the used PEM stack. For the specifically foreseen PEM stack (Schaeffler 30 cells stack) the maximum hydrogen production is 11 Nm³/h (about 1 kg/h). The maximum required hydrogen flow for the SEDMES (feed flow only, purge flow will be fed by the infrastructural hydrogen feed) is 14 Nm³/h. The maximum hydrogen production of the Nextgen is not enough to fulfill the SEDMES feed stream needs, therefore an infrastructural back-up of hydrogen is required.

Temperature: The maximum outlet temperature of the hydrogen from the Nextgen is 80 °C. This affected the choice of the compressor to compress the hydrogen to the needed pressure for the SEDMES. But by the demands for the humidity of the hydrogen, the gas will be cooled and dried before entering the compressor. The selected air driven hydrogen booster can handle gas up to 100 °C at the outlet, therefore the hydrogen gas at the inlet of the booster should have a temperature of 20 °C or lower.

To make sure both the Nextgen and the SEDMES can be started-up, operated and shutdown independently as well as combined with the other unit, a buffer for the H₂ was incorporated and control system was set-up to steer e.g. the source H₂ being sent to the SEDMES.

Operation: During continuous operation it should be possible to operate both the units without disturbing the operation of the other unit. During operation of the combined units several alignment problems can happen, which have to be tackled by the (control) system.

All required components were procured, the connection was constructed in Petten (Figure 5) and a control system was developed.



Figure 7: Pictures of the PEM-SEDMES connection. 1: Outside view showing connection between the nextgen container (light blue) and novel 10ft container with the hydrogen buffer. 2: Outside view with the connection between the SEDMES system and PEM. 3: Details inside and on the 10 ft container.

2.2.4 SEDMES unit improvements and further process integration

During the project and the first experimental campaigns with the SEDMES pilot (without the PEM electrolyser) at higher pressures (40 bar), several issues arose:

- Hydrogen leakages. Hydrogen is a very small gas and caused several leakages that had to be solved.
- Attrition of the sorbent material caused high pressure drops in the system. This was not observed in previous project on smaller scale installation. Therefore we had to replace some badgers and place filters under the reactors.

The downstream purification unit (DPU) has been significantly delayed at the supplier. In the project preparation were made to receive the DPU in Petten (e.g. basic design and preliminary engineering skid interconnect for SEDMES and DPU columns; lay-out study; purchase of containers for DPU coolers).

The issues mentioned above were solved and the SEDMES pilot installation was improved in terms of flexibility, stability, ease of maintenance and increased pressure to optimize productivity by:

- Addition of a cage ladder on the containers
- Doors in the reactor tower for ease of maintenance
- Flow indicators improved
- Sampling system improved
- 10ft containers for add-ons e.g. purification system

The PEM-SEDMES connection was realized and commissioned. A experimental testing plan (Table 1) was set-up for a month of 24/7 operation investigating several parameters, validating the simulations and proofing the P2DME concept. Unfortunately, this could not be realized within the timeframe of the project due to the issues described above and will be conducted in the follow-up projects iDME and POWERED.

Table 1: Experimental plan, SEDMES operated at 250 °C.

Experiment name	Pressure (bar)	ADS flow [Nm ³ /h]	Purge flow [Nm ³ /h]	# cycles	H2 source	Purpose
<i>Bench Mark (BM)</i>	20	13.3	20	10	Infra	Reference
Standard	20	13.3	20	10	Infra	
PEM20	20	13.3	20	10	PEM	Proof of concept P2DME
<i>BM</i>	20	13.3	20	10	Infra	Reference
short cycle	20	20	20	10	Infra	
<i>BM</i>	20	13.3	20	10	Infra	Reference
P30	30	13.3	20	10	Infra	
<i>BM</i>	20	13.3	20	10	Infra	Reference
PEM30	30	13.3	20	10	PEM	
<i>BM</i>	20	13.3	20	10	Infra	Reference
P30 short cycle	30	20	20	10	Infra	
<i>BM</i>	20	13.3	20	10	Infra	Reference

2.3 Market and outlook

The market potential for Power-to-X (PtX) technologies is promising, as the demand for high-value chemicals, such as methanol and dimethyl ether (DME), continues to grow steadily^v. Estimates suggest that the global market for PtX could reach up to €2 billion by 2035. Additionally, carbon capture, utilization, and storage (CCUS) technologies play a crucial role in reducing emissions in industries, and the EU has recognized their significance. The potential for CCUS to capture and store CO₂ emissions provides a valuable feedstock for PtX processes, which are poised to meet the rising demand for sustainable chemicals and e-fuels, particularly in sectors like chemical and steel industry, shipping, and aviation. However, the transition to sustainable production, access to low-cost green hydrogen, and enabling policies will influence PtX's competitiveness in the coming decades.

Moreover, with the increase in the price of EU Emissions Trading Scheme (ETS) credits, additional national funding, and carbon taxation policies, the number of CO₂ capture projects is expected to grow considerably in the EU. These projects, often focused on permanent geological storage, may transition to CO₂ utilization in Power-to-X applications if supportive policy and regulatory environments emerge. The shift from storage-focused CCUS to CO₂ utilization aligns with the broader effort to move towards more sustainable, circular carbon economy practices, providing a new avenue for PtX technologies to contribute to emissions reduction while meeting growing market demand for high-value chemicals and e-fuels in various transport sectors.

Renewable Dimethyl Ether (rDME) specifically offers a compelling solution to the evolving energy supply needs in the industrial sector. With an annual demand of 12.4 million tonnes for off-grid applications in the UK and EU, particularly for heating and drying processes, there is a substantial existing market. Additionally, 11.2 million tonnes of LPG are used for transport in the UK and EU. However, the reliance on fossil fuels for these applications is facing challenges due to evolving policies limiting fossil fuel utilization. rDME can serve as a blend of up to 20% in LPG, offering an immediate low-carbon alternative, or as a 100% rDME fuel with minor appliance modifications. This versatility positions rDME to potentially capture a significant share of the existing LPG market, especially in off-grid applications.

Furthermore, rDME plays a crucial role as a bulk platform chemical, with applications in aerosol propellants, solvents, and as a feedstock for ethylene production. Ethylene has a broad portfolio of applications, including industrial uses in metal fabrication, food and beverages, agriculture, refining, and plastics production. The annual demand for polyethylene (PE) and polypropylene (PP) in the EU in 2020 was 15 million tonnes and 10 million tonnes, respectively. This highlights the substantial role rDME can play in the production of common plastics, contributing to a more sustainable and renewable feedstock.

A recent technoeconomic evaluation of the P2DME SEDMES technology, at a scale of 23 kt/year, projected the production cost of approximately €1.3 per kg of DME.ⁱ Though currently higher than market prices, this cost positions it competitively with conventional CO₂-based DME synthesis costs. The evaluation shows that the primary cost driver is the expense of H₂. As such, the economics of SEDMES emphasize its efficiency in converting green H₂ into DME, making strides within the power-to-X sphere. The crucial factors impacting its viability remain the costs associated with electricity, CO₂, and notably, the capital expenditure linked to the electrolyzer—an integral component for technological advancement and cost reduction in the long run. When utilizing offshore wind power for H₂ synthesis and DME production, the cradle-to-grave carbon footprint of using DME as a transport fuel is 77% lower than petrodiesel.^v Furthermore, by incorporating renewable energy for CO₂ capture and utilizing waste heat in DME production and purification, the potential exists for DME's carbon footprint to exceed a 90% reduction compared to fossil fuels.

The SEDMES technology will be further developed in several follow-up projects. The POWERED project^{vi} aims to develop and optimize the P2DME process, utilizing renewable energy sources to convert CO₂ into valuable fuels and chemicals. The variable nature of wind-generated electricity creates flexibility needs within the entire production process of power-to-X technologies, such as electrolysis and DME synthesis. Fluctuating electricity prices due to availability further compound these challenges. To address this, POWERED aims to create an adaptable energy management system (EMS) that ensures essential flexibility and minimizes potential risks of system failure. In the project a purification system and storage will be added to produce DME meeting fuel standards outlined by ISO as well as chemical standards, and experimental campaigns at different locations will be performed to test application in different use cases (steel, oil and gas and process industry). Furthermore, the final objective is the concept

design of the demonstration plant, scaling up SEDMES technology from the pilot plant scale (TRL6) to demonstration scale (TRL7).

The Butterfly project focuses on the combination of biomass gasification and SEDMES. It aims to facilitate adaptable production of renewable DME and synthetic natural gas (SNG) using both biological and non-biological source materials. At TRL7, the project intends to showcase a versatile, cost-effective process merging indirect gasification with SEDMES, succeeded by methanation, to generate rDME and SNG. Last but not least, TNO is discussing follow-up testing projects in the nextgen container with Schaeffler.

3 Power2FA

3.1 Introduction to FA

Electrochemical reduction of CO_2 towards formic acid has been widely investigated on different metal electrodes. In order to form formic acid, the pH of the electrolyte must be below the pK_a of the formic acid (3.74). However, if the electrolyte has an acidic nature the competitive reaction, namely Hydrogen Evolution Reaction (HER) is favoured, thereby decreasing the selectivity towards formic acid. When the electrochemical reduction of CO_2 is performed in neutral or basic conditions, the selectivity towards formate is greatly enhanced. The formation of formate instead of formic acid leads to the need of a more complex Down Stream Process (DSP) where the formate ion first has to be protonated to formic acid. We propose a new concept for the in-situ generation of formic acid during electrolysis, which will decrease the complexity of the further DSP.

Conventionally, CO_2 electrolysis is performed in a press filter flow cell consisting in two compartments: anolyte and catholyte compartment, which are separated by an ion exchange membrane (See Figure 8a). Our approach consists of adding an extra compartment (middlelyte) in between anolyte and catholyte (See Figure 8b). In this configuration the anolyte is separated from the middlelyte by an anion exchange membrane, and the middlelyte is separated from the catholyte by a cation exchange membrane. In this three compartment configuration, the formate formed on the cathode diffuses through the anionic exchange membrane (AEM) to the middle compartment where it recombines with the protons that are generated on the anode side during oxygen evolution reaction which diffuse towards the middle compartment through a cationic exchange membrane (CEM).

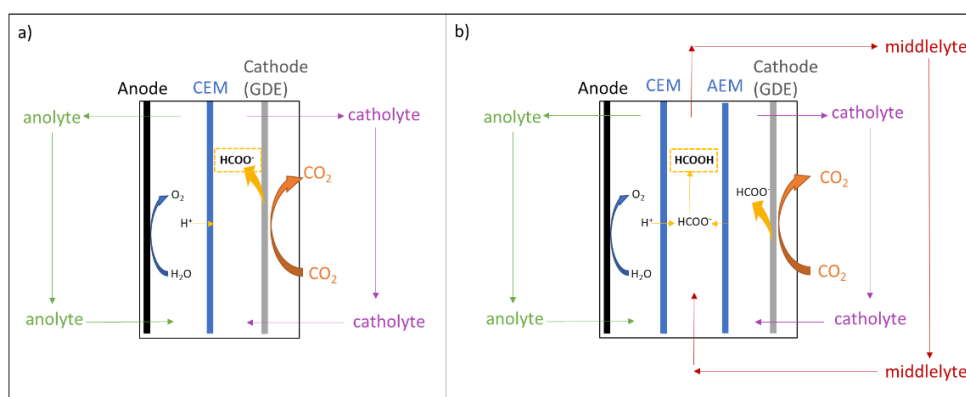


Figure 8. Schematic representation of the electrochemical reactor for CO_2 reduction to a) formate in a conventional two compartment cell and b) formic acid in a three compartment cell.

3.2 Technical report

3.2.1 ELEKTRA: 100 to 400 cm^2 scale

The ELEKTRA setup is a flexible and versatile bench scale electrolysis setup. The main functionality of ELEKTRA is the use of gas diffusion electrodes at ambient or elevated

temperatures and pressures for the reduction of CO_2 or CO . Of particular interest for this project is the electrochemical synthesis of formic acid from CO_2 and the possibility of in situ separation using a three compartment reactor. Figure 8 shows the reactor configurations aimed to be studied.

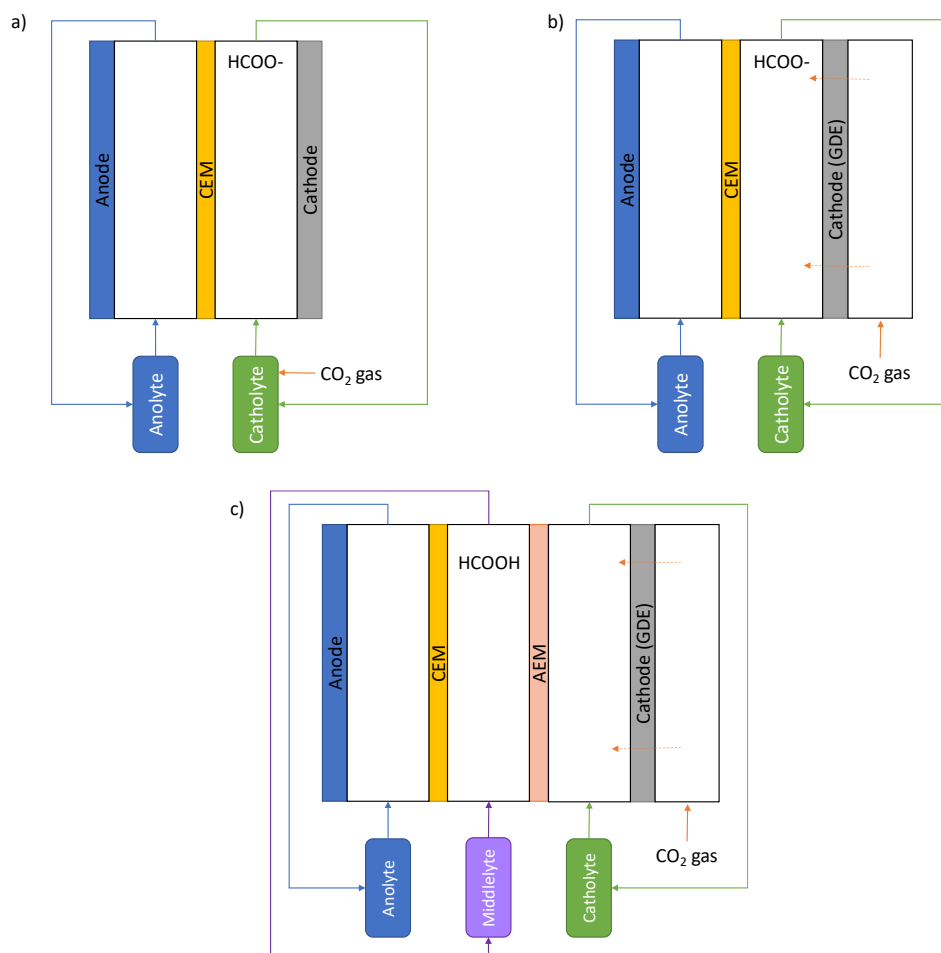


Figure 8: Tested reactor configurations in the ELEKTRA setup; a) Two compartment with CO_2 feed to the catholyte vessel; b) Two compartment with gas diffusion electrode (GDE) cathode and an extra chamber for feeding humidified CO_2 gas; and, c) Three compartment with GDE cathode and extra chamber. The product in configurations a) and b) is formate and in c) is formic acid.

The above configurations represent one cell pair with an active area of 100 cm^2 . By stacking this configuration, a stack reactor is achieved and the active area is increased. Figure 9 schematizes the stack reactor, and how the cell pairs connect to each other. In this particular case, only two compartments are used.

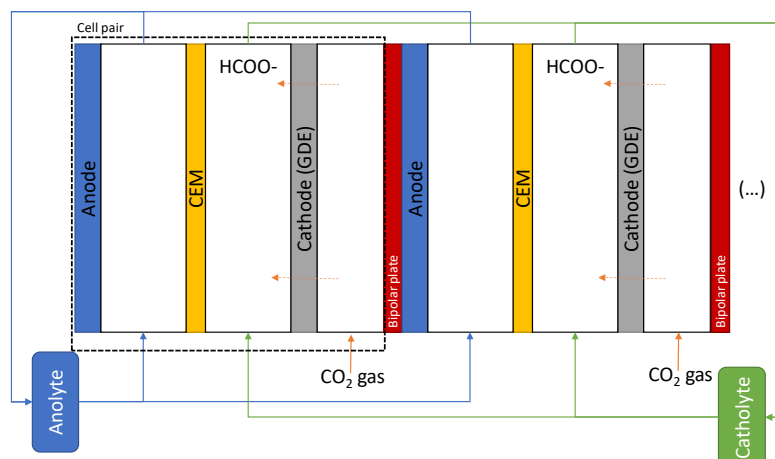


Figure 9: Stack reactor with two compartment configuration, showing two cell pairs connected by a bipolar plate.

On figure 10 and 11 photographs of both single and stack reactor are shown plus the setup ELEKTRA.

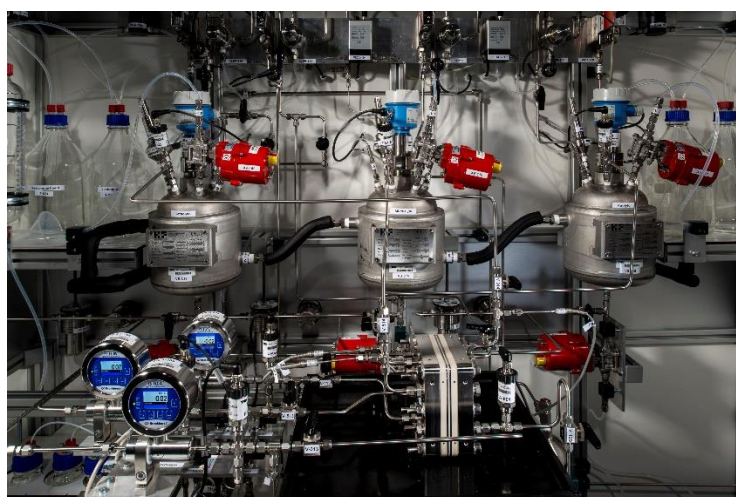


Figure 10: One cell pair electrochemical reactor used (middle bottom of the photo) and the overall ELEKTRA setup.

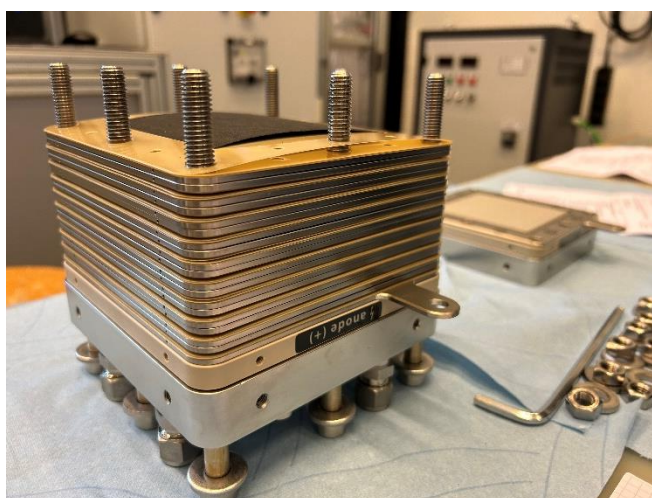


Figure 11: electrochemical reactor with 10 cell pairs during assembly.

The experimental work consisted in validating and comparing the different configurations and stack approach. Table 2 describes the constant parameters used in the experiments.

Table 2. Parameters used for the electrolysis of CO₂ to formate/formic acid.

Parameter	Description
Type of measurement	Chronopotentiometry
Cathode material	Sn plate or Sn-based GDE
Cathode size (cm ²)	100 per cell pair
Flow rate catholyte (L/h)	10
Membrane	Nafion 117 (2 compartment), AEM+CEM (3 compartment)
Anode material	Pt plate
Anode size (cm ²)	100 per cell pair
Flow rate of anolyte (L/h)	10
Flow rate of middlelyte (L/h)	10
Reactant	CO ₂
Delivery of reactant	In gas form, into the vessel, or directly to the back of the GDE
Cell pair number	1 or 4

Test 1: Configuration with two compartment with CO₂ feed to the catholyte vessel (Figure 12.a) single cell pair – electrolyte composition

The first test was used as a verification of the ELEKTRA after maintenance and suitability/compatibility of different electrolytes with the system. On the first part, the catholyte solution was 1 M KHCO₃ and the anolyte solution was 0.5 M H₂SO₄. Acidic environment at the anolyte enhances the formate production with higher availability of protons.

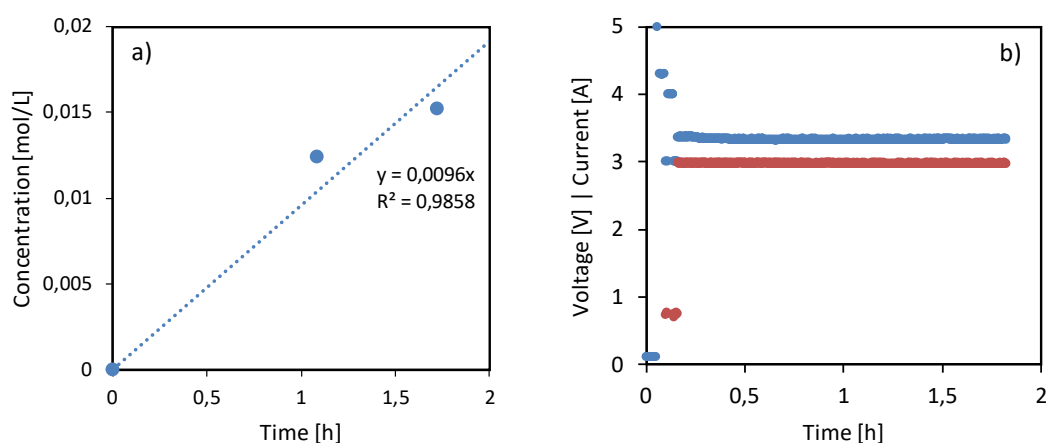


Figure 12. a) Concentration of formate at the catholyte over time; b) Systems voltage and current throughout the experiment.

The experiment was performed during two hours and formate production was detected at the catholyte, however the production rate was only 0.0096 mol.L⁻¹.h⁻¹ (Figure 12.a). The low production rate can be explained by the non-optimized conditions of this first experiment as

low current (Figure 12b), and by using two liquid compartments and feeding the gas to the vessel, which does not allow a good mixture of CO_2 with the solution. Furthermore in these two hours of experiment there was coloration of the anolyte solution (Figure 13), which indicates undesirable products appear.

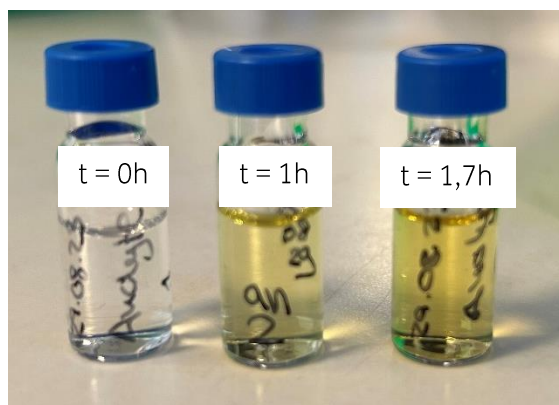


Figure 13. Coloration of anolyte during two hour experiment.

To further investigate the origin of the coloration these samples were submitted for Inductive Coupled Plasma (ICP) searching for Ti, Fe, Cr and Ni, as this could indicate corrosion of stainless steel, which is the main material on ELEKTRA (tubes, vessels, etc.). In addition, to the acidic environment at the anolyte (Figure 14a), a second part of the test consisted in doing it in an alkaline environment with 0.5 M KOH as anolyte and catholyte (Figure 14b).

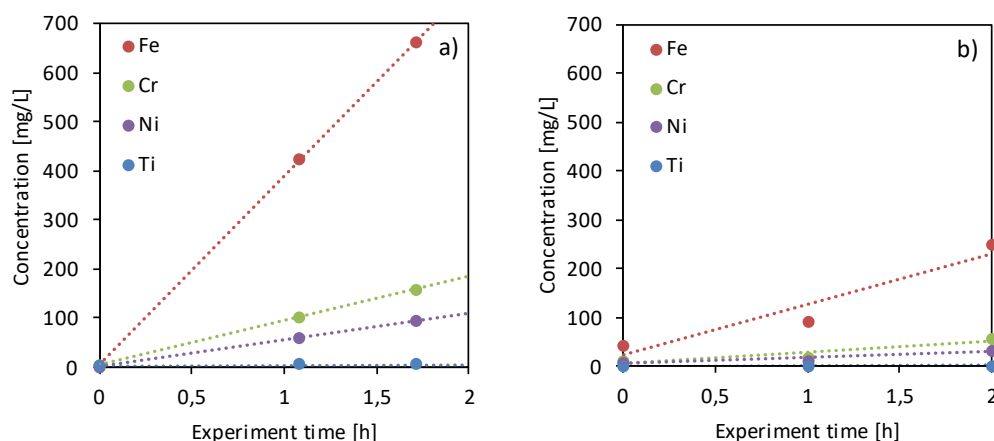


Fig 14. Results from ICP regarding Fe, Cr, Ni and Ti. a) experiment in acid environment; b) experiment in alkaline environment

The results presented in Figure 14 indicate that the presence of electrolysis and acid environment lead to leaching of metals in solution to almost a total of 1000 ppm (1 g.L^{-1}) after two hours. For the case of the alkaline environment with KOH, the increase of metals in solution is also seen over time, but four times lower the effect. It is concluded that acidic environments should be avoided in ELEKTRA to protect and avoid degradation of the setup. The catholyte compartment did not show change in coloration and therefore it was not checked at this point.

Test 2: Configuration with two compartment with CO₂ feed to the catholyte vessel (Figure x1a) single cell pair – evaluation of potassium bicarbonate as electrolyte

The cell was carefully rinsed between Test 1 and Test 2 to assure most metals dissolved in solution were removed. Using ICP it was obtained that the rising water from both anolyte and catholyte contained less than 1 ppm. The experiment was repeated with 0.5 M KHCO₃ in both anolyte and catholyte while keeping the other conditions similar to Test 1. One major difference was that at 1,5 hours the experiment was stopped for 3 days.

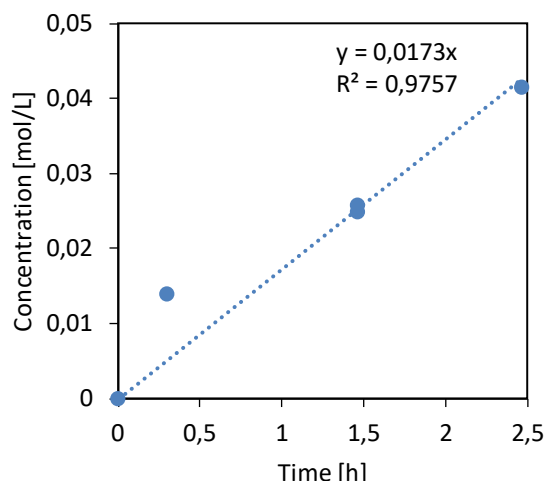


Figure 15. a) Concentration of formate at the catholyte over time. b) Systems voltage and current throughout the experiment.

The production rate of formate increase compared to Test 1 in almost twice the rate (Figure 15). At the same time the coloration was not visible in the samples obtained. This was further checked with ICP and the results were positive towards elimination of dissolved metals.

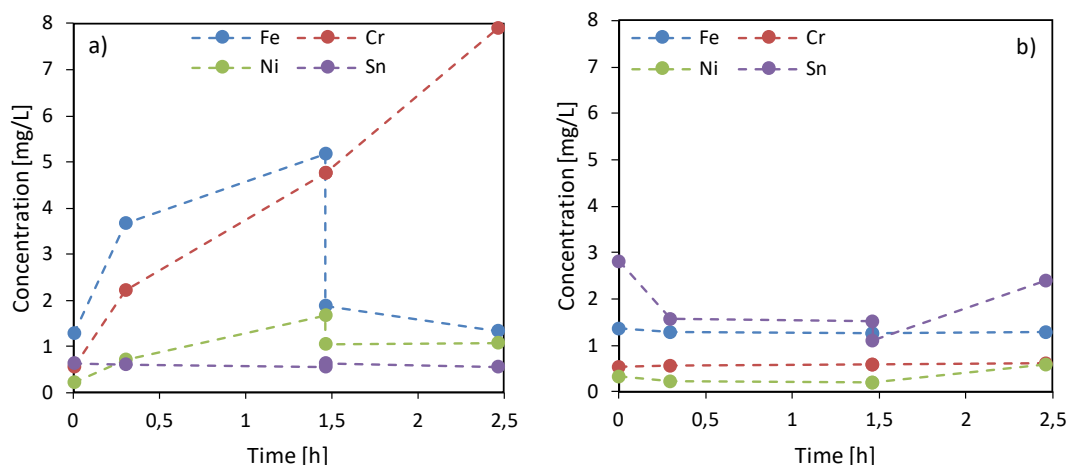


Figure 16. Results from ICP regarding Fe, Cr, Ni and Sn in a) anolyte and b) catholyte.

Figure 16 shows the concentration of Fe, Cr, Ni and Sn in both anolyte and catholyte. Sn was added to the searching metals since it is the cathode material and Ti did not show to be an issue in previous analysis therefore it was removed. It is important to note the scale of the y axis when comparing Figure 16a and Figure 14. The concentration of metals in the anolyte is

reduced to below 10 ppm which is 100 times less than what was obtained in the same experimental time frame previously. This indicates that the leaching of metals can be controlled by the use of neutral solutions like KHCO_3 . On Figure 16a, it is possible to see a distinct behaviour for the Fe at 1,5 hours, when the experiment was stopped for 3 days. It is hypothesized that with a relaxation time (no current applied) the Fe deposits at a surface in the loop. The catholyte shows a constant low amount of dissolved metals again confirming that no issues are found in this compartment.

Future testing and plans for ELEKTRA

Due to the challenging nature of operating a larger scale setup with issues, not only shown by the leaching of metals but also defected parts which took time to be replaced and make the system functional again, not all the goals were achieved. Here is discussed the further steps on ELEKTRA for the production of formate/formic acid.

The next testing shall focus on the configuration of two compartment with gas diffusion electrode (GDE) cathode and an extra chamber for feeding humidified CO_2 gas (Figure 1xb). Custom made GDEs of Sn from Fuel Cell Store have been purchased for this purpose. With this it is expected to increase the working current densities and the production rate of formate. Parameters like temperature, flow rate and pressure should be varied to find optimum working conditions, while keeping it feasible bigger scale processes. Once the concept is shown at a single cell for this configuration, the next step is to test a stack with 2 or more cell pairs to evaluate the scalability of the process by stacking (Figures 9 and 11). Lastly this approach may be compared to the configuration of three compartment with GDE cathode and extra chamber. Where the direct product is formic acid.

4.2.2. ZEUS: 400 to 6000 cm^2 scale

The ZEUS set up, presented in figure 17, is a large scale electrolyser designed with the capability of producing 1kg/h of formate via CO_2 reduction. The set up main differentiators are its size (6 m^2 of electrochemical surface area), its capacity for long term testing due to its feed-and-bleed operation, and its versatility, being able to function in different reactor configurations and across a broad range of operational conditions.

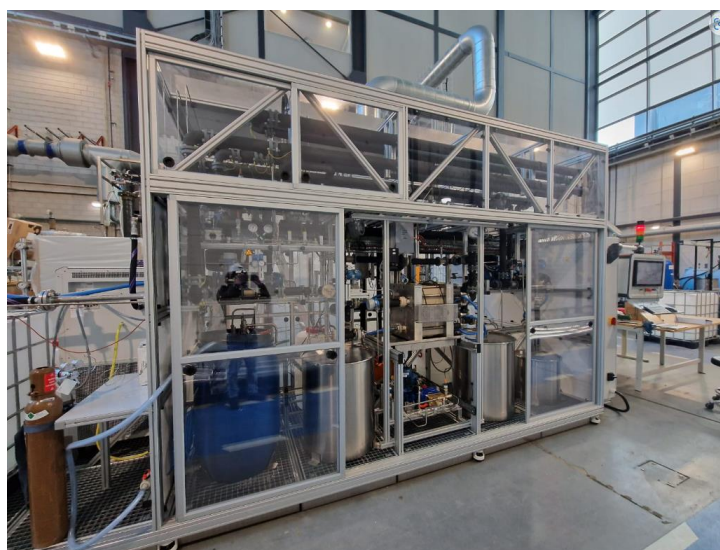


Figure 17. ZEUS Setup

In concept, the ZEUS setup will work in conjunction with ATHENA, a 2-15 cell reactor that allows for 2 types of configurations, as shown in figure 18

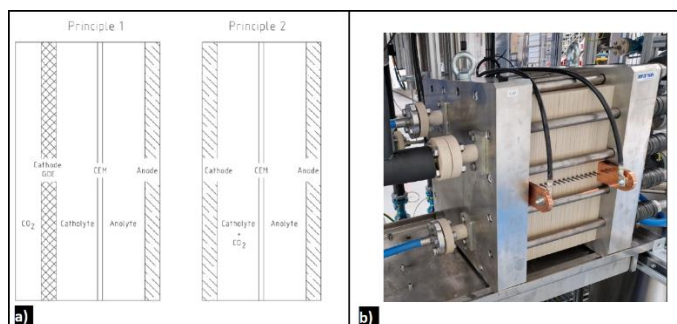


Figure 18. a) Modes of operation of Athena reactor. b) Athena reactor with full 15 cell stack

On configuration 1, a gas diffusion electrode (GDE) is used as cathode, with CO₂ moving through it from the backside into a liquid compartment wherein CO₂-free catholyte flows, with the triphasic gas-liquid-solid interface needed for conversion presenting as the bubbles travel through the GDE and contact the liquid flow. On the other compartment, liquid anolyte flows parallel to a platinized titanium anode. The 2 compartments are separated by a cation exchange membrane (CEM). On configuration 2 no GDE is used, and CO₂-rich catholyte flows parallel to a cathode plate, with the CO₂ molecules accessing its surface by diffusion inside the liquid. Once again, the catholyte compartment is separated from the anolyte compartment by a CEM. Which configuration to use is selected before the start of testing via the process control software, pictured in figure 19, and both configurations can be used across a range of pressures and temperatures, allowing to test their impact on the formate production rate.

Pressurization		
PICA-903: Main	1 Barg	Range: 0 - 20 Barg
Temperature control		
E-701: Temperature	20 °C	Range: 0 - 90 °C
TICA-102: Catholyte loop	20 °C	Range: 0 - 80 °C
TICA-402: Anolyte loop	20 °C	Range: 0 - 80 °C

Figure 9. Pressure and Temperature range of ZEUS operation

The ZEUS set up also allows for feed-and-bleed operation, allowing for long term experiments to take place. As the electrolysis reaction takes place, both anolyte and catholyte experience the following changes, among others:

1. Increase of formate concentration in catholyte due to CO₂ reduction
2. Alkalinization of catholyte due to OH⁻ formation during unwanted water reduction
3. Decrease of anolyte acidity as protons move through the CEM
4. Transfer of water between anolyte and catholyte compartments
5. Contamination of electrolytes due to impurities

This means that in order to maintain a stable operation during long term testing, both electrolytes have to be continuously regenerated. ZEUS allows for this by periodically withdrawing a certain amount of used electrolytes, and feeding new catholyte and anolyte into the system. When this renewal occurs can be chosen in the process control software, either after a certain amount of time, or after a certain amount of charge has been transferred, as shown in figure 20.

Figure 20. Selection screen for type and amount of drains of catholyte and anolyte.

4.2.2.1 ZEUS operation

The ZEUS set up operation is based on the sequenced activation of a series of subsystems, as presented in table 3:

Table 3: List of ZEUS subsystems and brief description of their features

SUBSYSTEM	FUNCTIONALITY
Safety Utilities	Emergency buttons, ventilation, H ₂ detection, O ₂ detection, CO detection, liquid leak detection. Complete depowering and shut down of set up if limits are reached
Catholyte Feed	Input of the KHCO ₃ catholyte into the preparation and catholyte vessel. Also included in this subsystem is the collection of the produced formate after the predefined set time/charge
Anolyte Feed	Input of the H ₂ SO ₄ catholyte into the anolyte vessel. Also included in this subsystem is the collection of the depleted H ₂ SO ₄ after the predefined set time/charge
Pressurization	N ₂ is fed through the electrolyte vessels into the system to increase the pressure up to the desired set value
Catholyte & Anolyte Loop	Catholyte and anolyte are circulated by the circulation pumps through the ATHENA reactor, into the heat exchanger, and back into the electrolyte vessels
Temperature Control	Water is flowed through the heat exchanger to cool down the warm electrolytes after passing through the ATHENA reactor
Carbon Dioxide Feed	CO ₂ is fed to the system. The gas flow is directed to the catholyte vessel or to the back of the GDE in accordance to the configuration selected before the start of the test, either "To GDE" or "To vessel" on the process control software
Power Supply	A fixed current or voltage is applied to the ATHENA reactor

ZEUS allows for the sequenced activation of these subsystems to occur both manually or automated, based on operator's choice in the process control software. Each subsequent system can only be accessed when the previous one is active.

4.2.2.2 Experimental data

Two experiments are planned, to answer the following research questions:

Q1. What operating conditions are feasible to be used in the pilot demonstrator and how to translate them from the lab-scale tests?

Q2. What are the maximum current densities and faradaic efficiencies towards formic acid that can be achieved during electrochemical conversion of CO₂ at larger scale?

Q3. How is the scalability of the process from 100 cm² to 400 cm²? How are the key performance indicators achieved during CO₂ electrolysis compared from lab-scale experiments (100 cm²) to larger scale experiments performed in the pilot demonstrator (400 cm² single cell)

Q4: Can we increase the production of formic acid by increasing the pressure of the system?

The experimental conditions in these 2 experiments are the following:

Fixed conditions	
Type of measurement	Chronopotentiometry
Cathode material	Sn-based GDE manufactured by VITO
Cathode size (cm ²)	400 (20 cm x 20 cm)
Catholyte	1 M KHCO ₃
Flow rate catholyte (l/h)	To be determined during preparation phase
Membrane	Nafion 117
Anode material	IrO ₂ GDE provided by MTSA
Anode size (cm ²)	400 (20 cm x 20 cm)
Anolyte	H ₂ SO ₄ 0.5M
Flow rate of anolyte (l/h)	To be determined during preparation tests
Reactant	CO ₂
Delivery of reactant	In gas form directly to the gas diffusion electrode without humidification
Flow rate of reactant (l/h)	To be determined during preparation tests
Analysis	Liquid samples and gas samples will be collected every hour and analysed by chromatography to determine the production of formic acid and side products.

3.3 Market outlook on formic acid

3.3.1 Market status

The global production of formic acid was less than 1 million ton per year back in 2015 and is expected to grow more than double by 2030, mainly as a result of its potential new applications as energy carrier and fuel. It is expected the formic acid market will grow from 1.4 billion euros in 2023 to 2.6 billion euros by the end of 2033 at CAGR with an average annual

production growth in a range of 3.8 to 4.9% on a global scale. Production capacity in Europe in 2009 was around 350 kta out of which 60% in Germany and 30% in Finland. Production capacity in Asia was around 370 kta, mainly in China. Production leaders in Europe are BASF in Germany (200 kta), Tamico (former Kemira) in Finland and Perstorp AB in Sweden. Other production leaders in the world are Eastman Chemical Company in USA, Luxi Chemical Group and Beijing Chemical Industry Group in China, and Gujarat Narmada Valley Fertilizers & Chemicals Limited in India. An outlook to global market growth till 2030 for formic acid is presented in Figure 1.

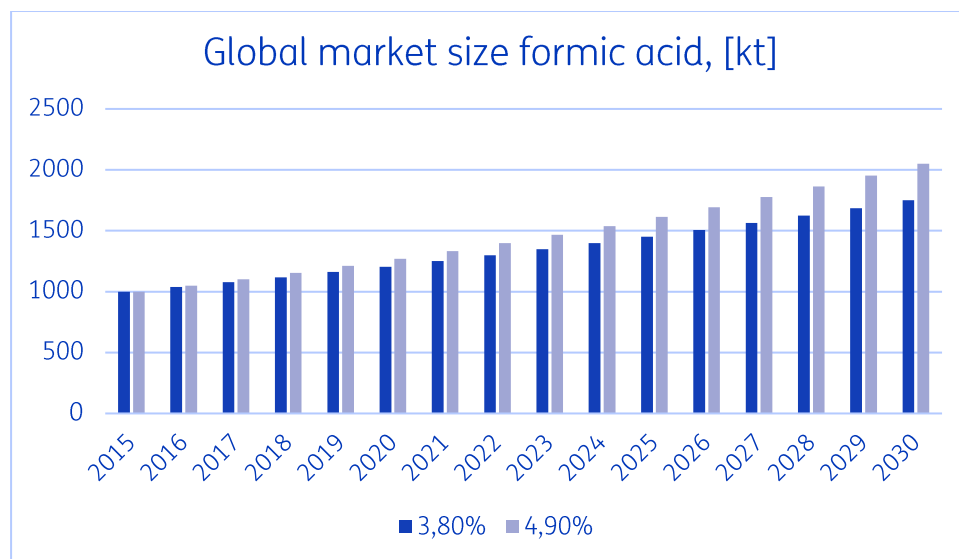


Figure 21. Market size for formic acid from 2015-2030

3.3.2 Application

Main applications of formic acid are in textile, rubber and pharmaceutical industry, followed by pesticides and dyes. Proven antibacterial properties expands its use in animal feed and as silage additive or preservative. It also can be used as a replacement for mineral acids like HCl and H₂SO₄ in steel pickling or in production of formate salts. Formate salts can be used for anti-freezing and as a cooling liquids. Formic acid is gaining interest as a carbon capture and utilization product, produced from CO₂, water and renewable energy. The research of a wide spectrum of its future possible applications like an energy vector, syngas storage medium, hydrogen gas carrier or carbon bio-source for different bioprocesses is being performed. Use of formic acid as a fuel is limited as there is a growing interest in using it as a hydrogen carrier due to easier transportation and storage than gas hydrogen.

Formic acid application

Animal feed

Textile industry

Rubber industry

Pharmaceutical industry

Hydrogen carrier

Syngas storage medium

Replacement mineral acids

One of possible applications of formic acid is also described by Voltachem, who produced formic acid and together with Team Fast tested its use in a fuel cell to run the city bus on electric energy. There is a plan to further investigate the use of formic acid as fuel for trucks, overseas transport and construction. The value chain presented by Voltachem is presented in Figure 22.

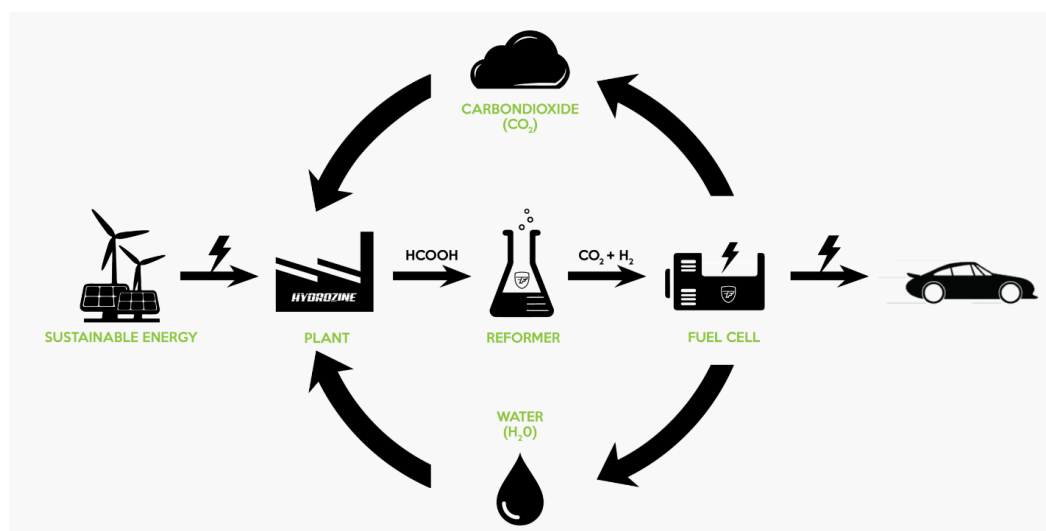


Figure 22. Value chain of sustainably produced formic acid

3.3.3 Technology implementation plan

A high-level technology implementation plan has been realized for the implementation of the CO₂ to formic acid conversion technology in the industrial scale.

The technology implementation plan consists of 4 phases as described in Figure . Phase 1 corresponds to TRL 2 of the technology where the idea is defined and the proof of concept is achieved. This phase is already finalized at TNO. Phase 2 corresponds to TRL 3-4 where the technology is validated at the laboratory scale. This validation at TRL 4 has been already achieved at TNO. Phase 3 corresponds to validation (TRL5) and demonstration (TRL6) of the technology at industrial relevant environment. This is the next phase that needs to be achieved, and it is envisioned to realize it by 2027. The final phase, Phase 4, corresponds to the prototype installation in the operational environment (TRL7) to demonstrate the

technology in the prototype level before the full commercial plant. The demonstration plant is targeted to be ready by 2030.

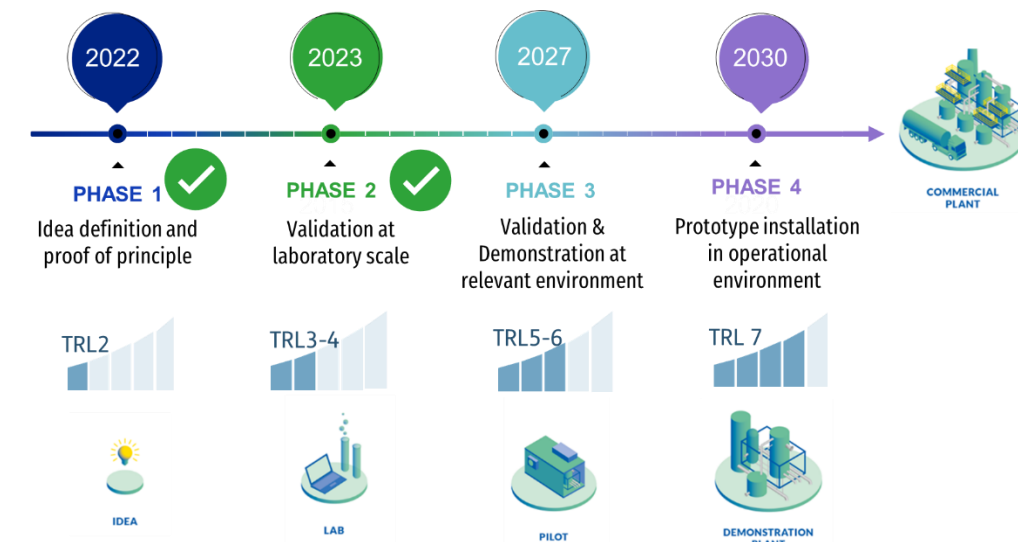


Figure 23. Phases of the technology implementation plan

For each of this technology development phases, a specific reactor area, stack and productivity of the final product (formic acid) was defined as shown in Figure 9.

The scale-up and scale-out process is as follows:

From Phase 1 to Phase 2: Scale-up from 0.001 m² to 0.01 m²

From Phase 2 to Phase 3: Scale-out from 1 cell to 5 cells stack with total area scale-up from 0.01 m² to 0.05 m²

From Phase 3 to Phase 4: Scale-up from 0.01 to 0.1 m² m

From Phase 4 to commercial plant: Scale-up from 0.1 to 1 m² m and scale-out from 5 cells stack to 100 cells stack with total area scale-up from 5 m² to 100 m²

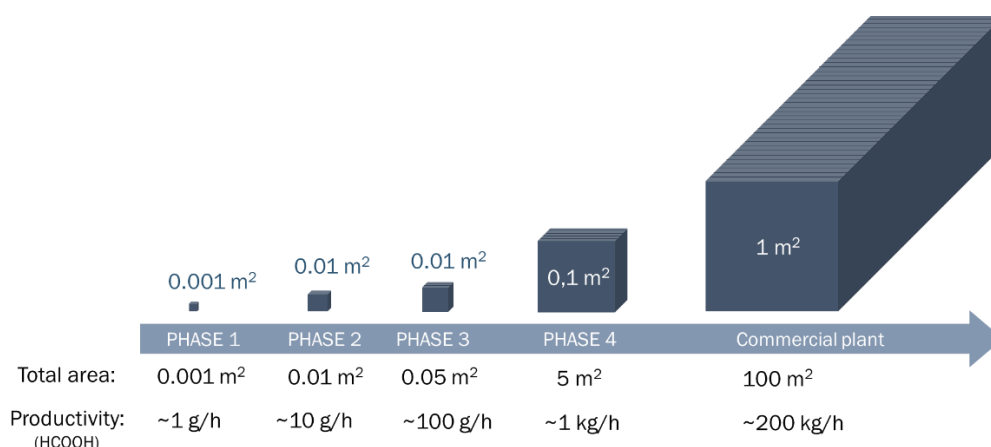


Figure 9. Single cell area and total area of electrolyser for the different phases of technology development, and targeted productivity of formic acid.

For the next phase (Phase 3) the activities defined for the technology development are as follows:

- **3.1 Technology scale-up (TRL 5)**
 - Electrolysis
 - Reactor design and optimisation towards zero-gap
 - Stack development and testing (5cells, 1000 cm²)
 - Downstream process
 - Development of down-stream processes (electrodialysis and azeotropic distillation)
- **3.2 Demonstration under relevant conditions (TRL-6)**
 - Long-term testing
 - Synthetic industrial-based feedstocks/streams
 - Validation using real process streams (RECYCLING LOOPS)
- **3.3 Integration of surrounding technologies with electrolysis**
 - Integration of CO₂ capture plant, fermentation process (feasibility studies and advanced techno-economic assessment)
- **3.4 Roadmap definition from TRL 6 to TRL 9**
 - Technology development plan for the complete plant to reach phase 3

3.4 Purification of formic acid and formate using renewable energy (Coval)

Introduction

Reducing CO₂ emissions and changing to renewable energy is more urgent than ever. In particular, the use of fossil fuels needs to be cut down, and in the meantime, the industry is facing the challenge of switching to processes using renewable energy. Sustainable production methods need to be developed, and the methods developed need to be at least price-competitive to accelerate the transition.

In literature, many methods are described for producing formic acid, formate, and more complex molecules using electrolysis and CO₂ as feedstock. However, the applicability of those methods at benchmark or pilot scale is not straightforward, as most research reports electrodes of limited size, i.e., 1-10 cm² (typical lab-scale experiments). Scaling up CO₂ electrolyzer to industrial size requires a major difference in design to both increase the surface area of CO₂ electrolyzer and stacking several hundred cells per reactor. In addition to the CO₂ electrolysis step, downstream purification of the obtained products is of most importance to (i) process the product up to commercial specification, both in concentration and purity, as well as (ii) minimize the energy consumption. As of now, a limited amount of papers describe the purification after an electrochemical reactor.

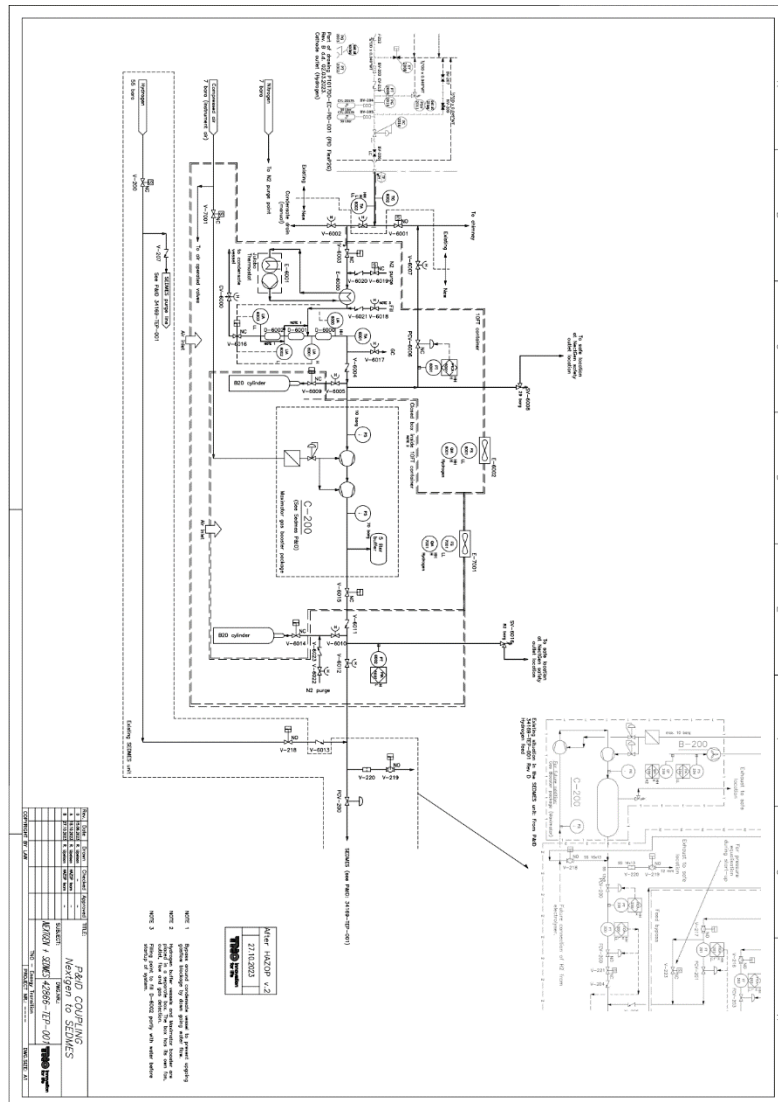
Project results

The in the Kansen voor West 2 project FLIE Coval worked to close the gap between the lab- and benchmark scale. Coval Energy focused on the downstream purification of formate obtained through CO₂ electrolysis from CO₂ gas. The least is known about this part of the value chain.

In the research project, various purification technologies were explored. Conclusions were drawn about technology potential, suitable electrolytes and optimisation of energy consumption. All purification techniques can achieve a formate concentration higher than one molar. The acquired data, results and generated IP are company confidential. The results open up the perspective for Coval to integrate the technology in a future product chain to convert CO₂ into sustainable aviation fuel.

4 Appendices

4.1 Appendix 1: P&ID PEM WE-SEDMES connection



5 References

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