Clean Hydrogen Partnership

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PEMTASTIC

Deliverable 1.3: Public report on definition of FC test protocols

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Content

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1. Executive summary:

This deliverable describes all test protocols and the defined test conditions to be used in PEMTASTIC. The test protocols are defined in terms of the cell voltage for tests in the differential cells and in terms of current density for short-stack measurements.

The protocols and the defined HD load profile enable the performance and durability investigation of the project MEAs using representative conditions for the HD truck application. The protocols and profiles will be used in WP4 for experimental durability investigations.

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2. Definition of FC test protocols

2.1. Test operating conditions for HD application

Representative test operating conditions for HD application are important to enable performance and durability characterization of the project MEAs. Different HD test conditions have been applied by different organizations as summarized in Table 1. Based on this summary, the PEMTASTIC consortium agreed on the “PEMTASTIC HD conditions” marked in yellow. The conditions are defined for stacks and technical single cells as well as for differential cells. For the latter, CEA calculated the conditions at stack air inlet and outlet based on a simple stack model using hydrogen and air in counter-flow and the coolant and air in co-flow configuration. The resulting anodic and cathodic pressure drop was calculated at a current density of 1.5 A cm^{-2}. These conditions (air inlet and air outlet) will be applied in the performance and durability tests and will be slightly modified for electrochemical characterization as stated in the following sections.

Table 1: HD test condition summary

Proposed by	DLR	IRD							CEA		PEMTASTIC HD condtions	
Source	internal	Ballard	Nikola(1)	Nikola(2)	Cummins	M2FCT(1)	M2FCT(2)	M2FCT(3)	internal	iDWG	At air **inlet**	At air **outlet**
Comment							AST	Benchmark	middle of stack	fixed flow 50 cm² U: 0.925 - 0.675 V		(calc by CEA)
Differential Cell												
Cell temperature [°C]	90	80	90	95	110	88	90	80	95	90	90	105
Gas composition	H2/Air	H2/air	H2/air	H2/air	H2/air	H2/air	H2/air	H2/air	H2/(N2+13%O2)	H2/air	H2/air	H2/(N2+9%O2)
Outlet pressure anode / cathode [bar_{abs}]	2.0/2.0	2.5/2.5	2.5/2.5	2.5/2.5	2.5/2.5	2.5/2.5	2.5/2.5	2.5/2.5	2.5/2.5	2.5/2.5	2.5/2.5	2.6/2.2
Gas inlet temperature anode/cathode [°C]	t_{cell} + 5°C								t_{cell} + 5°C		Cell temperature + 5 °C	
RH anode / cathode [%]	50/50	100/100	40/65	30/30	50/50	40/40	40/40	100/100	60/30	40/40	80/50	35/60
H2 and O2 stoichiometry for 4 cm channel length [-]	10/10								10/10		10/10	
Fixed gas flow according to current density [A/cm²]	3.0								3.0		3.0	
Stack / technical single cell												
Coolant inlet temperature [°C]											90	
Gas composition											H2/air	
Inlet pressure anode / cathode [bar_{abs}]											2.6/2.5	
Gas inlet temperature anode/cathode [°C]											95	
RH anode / cathode [%]											50/35	
Stoichiometry integral cell / stack [-]		1.5/2.0	1.5/2.0	1.5/1.8		1.5/2.0	1.5/2.0	1.5/1.8		2.9/2.9	1.2/1.8	
Fixed gas flow according to current density [A/cm²]										1.0	0.2	

All fuel cell tests in differential cells will be realized controlling the cell potential to assure comparability between the different test facilities and improve reproducibility. Only the short stack tests will be realized by current control.

2.2. Leak Test

A leak test will be performed after cell assembling as well as during BoT, MoT (after 500 h in longer durability tests) and EoT characterization of the durability tests. The leak test will be realized using the pressure loss method and the procedure harmonized by JRC for automotive application will be applied[1]. Nitrogen with a constant flow of 1 Nl/min (or 60 Nl/h) will be used to pressurize anode and cathode. The detailed procedure is shown in Figure 1.

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Objectives:

- Measure leakage rate by pressure decrease in anode and cathode compartment;
- Assure gas tightness of cell and test station.

Procedure:

1. Start dry nitrogen flow in anodic and cathode compartment at minimum flow rate;
2. Pressurize the anode compartment to 320 kPa (abs) and the cathode compartment to 300 kPa (abs). The pressure difference between anode and cathode (20kPa) should be the same as for the reference operating conditions;
3. Reduce nitrogen flow to zero;
4. Keep cell at this state for minimum 10 minutes;
5. Measure pressure decrease in both compartments for minimum 10 minutes;

Data analysis:

Plot anode and cathode pressure versus test duration for both situations when no cell is used and when the cell is used to determine the influence of the test bench gas tightness.

Acceptance criterion: the test is passed if the leak rate is lower than 0.3 kPa/min.

Figure 1: EU-harmonized leak test protocol for automotive applications[1]

2.3. Break-in

The MEA break-in procedure is applied to each pristine MEA to assure full performance of the MEA and to overcome initial, non-linear degradation, which might negatively impact the analysis of the degradation behavior of the MEAs. The procedure is defined by potentiostatic cycling based on the recommendations by IRD and adapted to the use in differential cells. Furthermore, the procedure includes H_2 and air soak steps for recovery of reversible degradation effect. These steps are adapted from the DoE recovery protocol[2]. Polarization curve measurements are included in the protocol to evaluated the break-in progress. Due to the different steps, the test conditions vary during the break-in procedure as shown in Figure 2. However, the conditions are generally adapted from "PEMTASTIC HD conditions" at air inlet (see Table 1) with increased relative humidity (90%) to assure membrane humidification and conditioning. The break-in procedure includes a final recovery step before BoT characterization and before start of the durability test.

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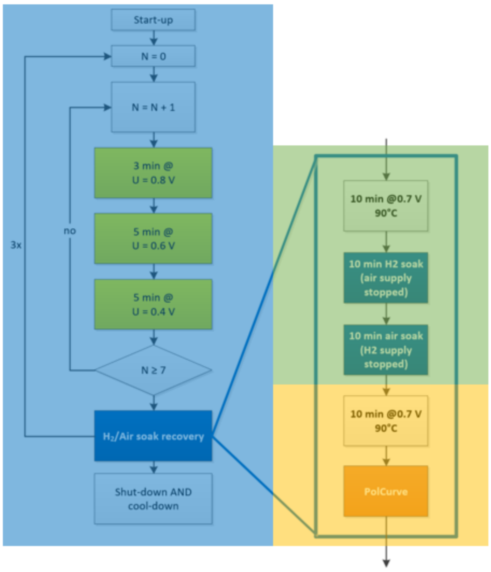
CYCLING	Conditions
Cell temperature [°C]	90
Gas composition	H_2/Air
Pressure anode / cathode [bar_{abs}]	2.5/2.5
Gas inlet temperature anode/cathode [°C]	95/95
RH anode / cathode [%]	90/90
H_2 and O_2 stoichiometry for 4 cm channel length [-]	10/10
Fixed gas flow according to current density [A/cm²]	3.0



SOAK RECOVERY	Conditions
Cell temperature [°C]	90
Gas composition	H_2/Air
Pressure anode / cathode [bar_{abs}]	1.5/1.5
Gas inlet temperature anode/cathode [°C]	95/95
RH anode / cathode [%]	90/90
H_2 and O_2 stoichiometry for 4 cm channel length [-]	10/10
Fixed gas flow according to current density [A/cm²]	3.0
Load has to be disconnected during soak time	

POL CURVE	Conditions
Cell temperature [°C]	90
Gas composition	H_2/Air
Pressure anode / cathode [bar_{abs}]	2.5/2.5
Gas inlet temperature anode/cathode [°C]	95/95
RH anode / cathode [%]	80/50
H_2 and O_2 stoichiometry for 4 cm channel length [-]	10/10
Fixed gas flow according to current density [A/cm²]	3.0

Figure 2: Break-in procedure in PEMTASTIC

2.4. Shut-Down and Start-Up

Shut-Down (SD) and Start-Up (SU) procedures are defined to enable reproducible cell start and stop for performance characterization, but also during the durability cycles, which includes several breaks (see section 2.9). The procedures are design to be representative for system operation and do not use nitrogen. Thereby, the stops are defined depending on the stop duration:

- **Short-stop: stop duration ≤ 30 min**
  - Pressure reduction to ambient pressure and load disconnected
  - Stop air flow (small hydrogen flow maintained)
  - Stop up to 30 minutes without cell cooling
- **Long-stop: stop duration > 30 min**
  - Pressure reduction to ambient pressure and load disconnected
  - Stop air flow (small hydrogen flow maintained)
  - Stop hydrogen flow (U < 200 mV, 10 min)
  - Cell cool-down

The shut-down procedure is composed of the following steps (steps 5-8 only for long-stop >30min):

1. Reduce load to 0.2 A/cm² or 0.8 V.
   -> Load ramp: 1.5 mA/(cm² s) or 1 mV/s
2. Reduce reactant pressures to ambient.
   -> Pressure ramp: 10 mbar/s
3. Reduce current to 0.05 A/cm² or 0.9 V and disconnect load.
   -> Load ramp: 1.5 mA/(cm² s) or 1 mV/s
4. Maintain small hydrogen flow of 10 mL/(min*cm²) at anode and stop air supply to cathode.
5. Stop humidification (bypass bubblers and maintain bubbler temperature)
6. Cool-down cell

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-> Cooling ramp: 2 °C/min

7. Maintain hydrogen flow for 10 min (cell voltage should be <200 mV).
8. Stop hydrogen supply

The start-up procedure is composed of the following steps:

1. Start coolant circulation and deactivate humidification (bypass bubblers).
2. Start hydrogen/air supply: purge cell using high flow rates to remove water and gas residues (differential cell flow rates: H_2 and O_2 stoichiometry of 10 for 4 cm channel length at 3.0 A/cm²).
   -> cell voltage check (> 900 mV)
3. Increase reactant (absolute) pressure stepwise to 1.5 bar.
   -> Pressure ramp: 10 mbar/s
4. Ramp-up load to 0.2 A/cm² or 0.8 V.
   -> Load ramp: 1.5 mA/(cm² s) or 1 mV/s
5. Increase reactant pressure to set points.
   -> Pressure ramp: 10 mbar/s
6. Ramp-up temperature
   -> Heating ramp: 2 °C/min
7. Activate humidification as soon as cell temperature > dew point (bubbler temperature)
   -> avoid flooding at any time!

It should be noted that the defined ramps for temperature, load and pressure have to be validated on the different test stations. If needed, the ramps will be adapted to match the possibilities of all test benches and to assure reproducibility and comparability.

2.5. Polarization Curve

The polarization curve procedure is also following the EU-harmonized protocol for automotive applications[1]. This procedure is defined regarding current density and can be directly applied for the small stack measurements in PEMTASTIC. For the differential cell measurements, the procedure is adapted and defined in cell voltage. Both tests require about 54 min of test duration (Table 2). The "PEMTASTIC HD conditions" at air inlet (see Table 1) are applied for this test.

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Table 2: Set points for polarization curve measurements in PEMTASTIC

Small stack measurements

Set point no.	Current density / A cm^{-2}	Recommended dwell time / s	Recommended data acquisition time / s
1	0.00	60	30
2	0.02	60	30
3	0.04	60	30
4	0.06	60	30
5	0.08	60	30
6	0.10	60	30
7	0.20	120	30
8	0.30	120	30
9	0.40	120	30
10	0.60	120	30
11	0.80	120	30
12	1.00	120	30
13	1.20	120	30
14	1.40	120	30
15	1.60	120	30
16	1.80	120	30
17	2.00	120	30
18	1.80	120	30
19	1.60	120	30
20	1.40	120	30
21	1.20	120	30
22	1.00	120	30
23	0.80	120	30
24	0.60	120	30
25	0.40	120	30
26	0.30	120	30
27	0.20	120	30
28	0.1	60	30
29	0.08	60	30
30	0.06	60	30
31	0.04	60	30
32	0.02	60	30
33	0	60	30

Differential cell measurements

Set point no.	Cell voltage / mV	Recommended dwell time / s	Recommended data acquisition time / s
1	OCV	60	30
2	900	60	30
3	880	60	30
4	860	60	30
5	840	60	30
6	820	60	30
7	800	120	30
8	750	120	30
9	700	120	30
10	650	120	30
11	600	120	30
12	550	120	30
13	500	120	30
14	475	120	30
15	450	120	30
16	425	120	30
17	400	120	30
18	425	120	30
19	450	120	30
20	475	120	30
21	500	120	30
22	550	120	30
23	600	120	30
24	650	120	30
25	700	120	30
26	750	120	30
27	800	120	30
28	820	60	30
29	840	60	30
30	860	60	30
31	880	60	30
32	900	60	30
33	OCV	60	30

2.6. Cyclic and Linear Sweep Voltammetry

Two sets of operating conditions are defined for cyclic voltammetry (CV) and linear sweep voltammetry (LSV) as shown in Table 3. The “standard conditions” are defined at 30 °C and atmospheric pressure, while the “HD air inlet conditions” are adapted from the “PEMTASTIC HD conditions” at air inlet (see Table 1).

Table 3: Test conditions for CV and LSV

	Standard conditions	HD air inlet conditions
Cell temperature [°C]	30	90
Gas composition	H_2/N_2	H_2/N_2
Pressure (absolute) anode / cathode [bar]	atm/atm	2.5/2.5
Gas inlet temperature anode/cathode [°C]	35/35	95/95
RH anode / cathode [%]	100/100	100/100
Gas flow H_2/N_2 [mL/(min*cm²)]	10/10	10/10

For both conditions the CV characterization is realized as following:

- Conditioning in H2/N2: until U_{cell} < 150mV, but at least 30 min
- Scan rate: 50 mV/s
- Potential range: 0.1 – 0.6 V
- Number of cycles: 3
- ECSA calculation: integration of H-desorption peak (base line defined following double layer capacity)

LSV characterization is realized mainly using the same conditions and parameters as used for CV, but differs in two parameters:

- Potential range: 0.05 V – 0.6 V
- Scan rate: 1 mV/s

The possibility to use non-flow configuration for CV and LSV will be elaborated in the protocol validation phase.

2.7. Electrochemical Impedance Spectroscopy

Electrochemical Impedance Spectroscopy (EIS) will be used in differential cells for more detailed analysis of the project MEAs. For this purpose, potentiostatic EIS is applied using different conditions.

H_2-Air-EIS is used to analyze MEA behavior in an operating fuel cell and the test conditions are the agreed "PEMTASTIC HD conditions" at air inlet as defined in section 2.1 and listed in Table 4. Additional parameters:

- Frequency: 20 kHz – 0.1 Hz
- DC potentials (Amplitude) in mV: 850 (2), 800 (2), 750 (5), 700 (5), 600 (5)
- Conditioning time: 10 min

Table 4: Test conditions for H_2-Air-EIS

	H_2-Air-EIS
Cell temperature [°C]	90
Gas composition	H_2/Air
Pressure (absolute) anode / cathode [bar]	2.5/2.5
Gas inlet temperature anode/cathode [°C]	95/95
RH anode / cathode [%]	80/50
H_2 and O_2 stoichiometry for 4 cm channel length [-]	10/10
Fixed gas flow according to current density [A/cm²]	3.0

H_2-N_2-EIS is used to determine the effective proton conductivity and media flow is adapted as listed in Table 5. Additional parameters:

- Frequency: 20 kHz – 10 Hz
- DC potential (Amplitude) in mV: 400 (5)
- Conditioning time: 10 min

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➔ Hydrogen crossover measurement during last 5 min

Table 5: Test conditions for H_2-N_2-EIS

	H_2-N_2-EIS
Cell temperature [°C]	90
Gas composition	H_2/N_2
Pressure (absolute) anode / cathode [bar]	2.5/2.5
Gas inlet temperature anode/cathode [°C]	95/95
RH anode / cathode [%]	80/50
Gas flow H_2/N_2 [mL/(min*cm²)]	10/10

2.8. Limiting Current Analysis

Limiting Current Analysis (LCA) is used in PEMTASTIC by CEA to analyze the different oxygen transport mechanisms in the cathodic catalyst layer. The tests will be realized in combination with the durability tests at BoT and EoT for the different project MEAs. Separate MEAs will be used for the BoT characterization to avoid the impact of potential cell degradation during the LCA tests.

The test conditions for the LCA measurements are summarized in Table 6. Additional parameters:

- Method:
  - Potentiodynamic scan: OCV -> 0.2 V
  - Hold 5 min at 0.2 V
- Variable parameters:
  - $c(O_2)$ in vol.-%: 0.50, 0.75, 1.00, 1.25, 1.50, 2.00, 2.25, 2.50
  - Anode and cathode (absolute) pressure in bar: 1.0, 1.5, 2.0, 2.5

Table 6: Test conditions for LCA

	H_2-N_2-EIS
Cell temperature [°C]	90
Gas composition	H_2/O_2 in N_2
Gas inlet temperature anode/cathode [°C]	95/95
RH anode / cathode [%]	80/80
RH anode / cathode [%]	80/50
Anode and cathode flow (CEA cell) [Nl/h] *	60/100

*: in CEA differential cell

2.9. HD Load Cycling Durability

The HD load cycle is developed by SYMBIO and DLR based on A VECTO-based HD mission profile of 11 h and 45 min including 9 h of driving, 2 h loading goods and 45 min break. It is described in "D1.1: Analysis of HD mission profiles". The final HD load cycle is defined in cell voltage for differential cell

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measurements and in current density for small stack measurements as shown in Figure 3. The profile is available for all partners via the project team site.

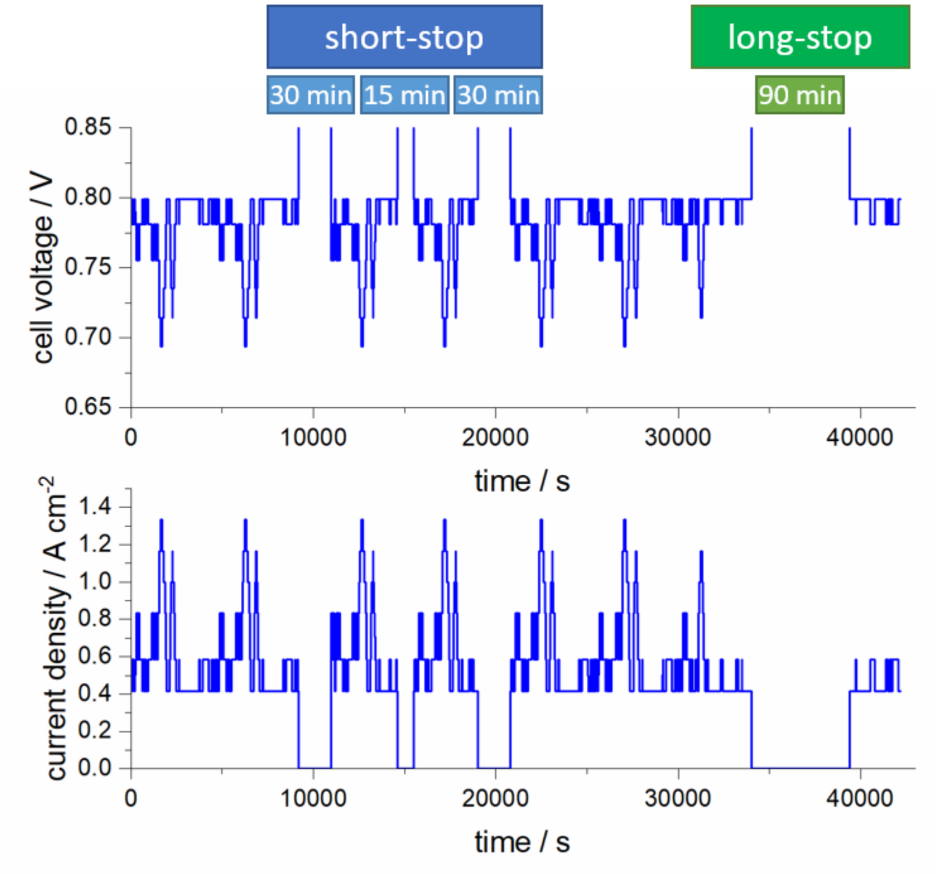

Figure 3: Developed load profiles for differential cell and small stack

The durability tests in the differential cells are realized in fixed flow mode and only the cell voltage is varied according to the load profile. The measurements in the small stack will be realized in constant stoichiometry mode and media flow has to be adapted as well. Furthermore, four start-stop-phases are included in the cycle, three short-stops (15 min and 2x30 min) and one long-stop (1.5 h). SD- and SU-procedures for these phases are defined in section 2.4.

3. Potential adaption

All defined protocols will be validated by all partners using their specific test setup and test station. If needed, the protocols will be adapted to match the possibilities of all test benches and to assure reproducibility and comparability. Furthermore, the operando stressor test protocols will be defined after the evaluation of the capabilities of the different test stations. These protocols are needed for the evaluation of the Gen1-3 MEAs and have to be defined before M9. Updated versions of the deliverables D1.2 and D1.3 will be provided as soon as all details are validated and reviewed.

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4. Conclusions

All required test protocols for PEMTASTIC are defined including Leak-Test, Break-In, Shut-Down and Start-Up, Polarization Curve, Cyclic and Linear Sweep Voltammetry, Electrochemical Impedance Spectroscopy, Limiting Current Analysis, and HD Load Cycling Durability. Additionally, the test conditions are defined. They are representative for conditions at air inlet and air outlet of a typical PEMFC stack for HD applications.

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5. Abbreviations, glossary, references, etc.

Abbreviations

BoT	Begin-of-Test
CV	Cyclic Voltammetry
DC	Direct Current
ECSA	ElectroChemically active Surface Area
EIS	Electrochemical Impedance Spectroscopy
EoT	End-of-Test
HD	Heavy-Duty
LCA	Limiting Current Analysis
LSV	Linear Sweep Voltammetry
MEA	Membrane Electrode Assembly
MoT	Mid-of-Test
PEMFC	Polymer Electrolyte Membrane Fuel Cell
RH	Relative Humidity
SD	Shut-Down
SU	Start-Up
VECTO	Vehicle Energy Consumption calculation TOol

References:

[1] G. Tsotridis, A. Pilenga, G. De Marco, T. Malkow. EU harmonised test protocols for PEMFC MEA testing in single cell configuration for automotive applications. https://publications.jrc.ec.europa.eu/repository/bitstream/JRC99115/ldna27632enn.pdf
[2] https://www.energy.gov/sites/default/files/2017/05/f34/fcto_myrdd_fuel_cells.pdf