# VOLUMETRIQ

## VOLUme Manufacturing of PEMFC Stacks for TRansportation and In-Line Quality Assurance

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# DELIVERABLE REPORT

| D2.3 – Stack component test scope and procedures | |
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| R | *Report* | X |
| P | *Prototype* | |
| D | *Demonstrator* | |
| O | *Other* | |

| SUMMARY | |
|---|---|
| **Keywords** | *Test procedures, use cases, operating conditions* |
| **Abstract** | *This deliverable concerns test scope and test procedures corresponding to the general requirement specifications as described in D2.1. The goal is to ensure repeatability of test results and to define representative operating conditions and test protocols. This will close the loop from stack specification to test verification. The test results can then be compared to the initial set of requirements and prove the compliance of the product. Test conditions will be delivered for the following operating conditions:*<br><br>1. *Test conditions for polarisation curves*<br>2. *Leak test procedure*<br>3. *Freeze start sequence*<br>4. *Start –up and shut-down sequence*<br>5. *Drive cycle as reference conditions for durability tests*<br><br>*Other, safety related test requirements, will be treated elsewhere as for example described in ISO-Standards or OEM test standards.* |

| REVISIONS | | | |
|---|---|---|---|
| **Version** | **Date** | **Changed by** | **Comments** |
| **0.1** | 01st February 2016 | Z. Veziridis, BMW | Draft version |
| **0.2** | 17th February 2016 | M. Armstrong-Brown | Additions to freeze start, break-in procedure, adaptions regarding EC cooling |
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| **0.4** | 24nd February 2016 | M. Armstrong-Brown | Revised Start-up/Shut-down, Cell Break-in |
| **1.0** | 25th February 2016 | Z. Veziridis | Released version |

# STACK REQUIREMENTS PROVIDED FOR FURTHER COMPONENT ANALYSIS

## CONTENTS

## ABBREVIATIONS

BoL – Beginning of Life
BoT – Beginning of Test
EIS – Electrochemical Impedance Spectroscopy
EoT – End of Test
FC – Fuel Cell
FC-DLC – Fuel Cell Dynamic Load Cycle
FSS – Full Scale Stack
NEDC – New European Drive Cycle

# 1 INTRODUCTION

The fuel cell stack test requirements described herein are the basis for proving the compliance of the product with the requirements specification, following defined and reproducible conditions. If the test results are positive it is assumed that the fuel cell stack will operate accordingly in a fuel cell vehicle and meet the target performance, dynamic behaviour and lifetime as specified. The test and validation of sub components will take place in the facilities of component manufacturers (JM) and stack integrator (IE). Single cells will be used to primarily confirm performance targets in a 50 cm² active area test set up for the performance target of 2,0 A/cm² at 0,6 V. Short stacks will be used to confirm the performance target of 2,5 A/cm² at 0,6 V as well as the capability to meet automotive drive cycle durability. There will also be a task to test a full size stack of 90 kW continuous and 120 kW peak power that also meets the target of 2,5 A/cm² in the proposed FC system configuration. Test requirements will be defined for

- Durability tests at full size short stack level and freeze start capability (e.g. 20 cell short stack)
- Performance tests at full scale stack (FSS) level proving 90 kW continuous and 120 kW peak power.
- Freeze start capability will be proven either with short stack tests or, if available in time, with FSS

Screening tests using single cells will be described in the report D2.4.

# 2 SCOPE

The scope of this report is to define test conditions for the relevant use cases that dominate the vehicle properties. The following operating states will be further detailed:

- **Start-up** sequence ("Key-on"): The fuel cell stack shall be operated from the "parking mode", where air and nitrogen are present in the anode and cathode compartment, to a normal operation mode. It is important to manage the media feed in such a way that the impact on degradation is minimal. The temperature range for the start-up might vary from (-) 25°C to (+) 85°C. Depending on the starting temperature, it will be distinguished between freeze start, cold start and warm start.

  - **Freeze start** starts at a stack temperature below 0°C. The specific requirement was set at (-) 25°C. The time necessary to reach 50 % of the nominal power (90 kW) after "key-on" shall be measured.

  - **Cold start** temperature range lies between 0 °C and 25 °C. The time necessary to reach 90 % of the nominal power after "key-on" shall be measured.

  - **Warm start** temperature range lies between 25 °C and 85 °C. The time necessary to reach 90 % of the nominal power after "key-on" shall be measured.

- **Normal operation:** After a successful start-up sequence, the stack supplies electric power according to a specific power demand. Characterisation of the overall performance is done mainly by polarisation curves in a defined range of temperature, pressure, air/hydrogen stoichiometry and humidity. The minimum cell voltage shall not fall below 0.35 V. The power transients can be as high as 100 kW/s. Normal operation includes different power modes like peak power (120 kW for 30 sec), nominal continuous power (90 kW) and minimum power (~8 kW at 0.85 V per cell). Below the minimum power demand, the fuel cell stack is going to a "stop mode".

- **Stop mode**: During this mode there is no power demand from the fuel cell stack, the anode compartment is a $H_2$ atmosphere and there is no air flow on the cathode side. If this mode is maintained for longer than 40 sec, then the shut-down procedure will be started.

- **Shut-down**: During the shut-down procedure the stack will be purged and prepared for a possible re-start. Excess water must be removed to enable a freeze start, in case the temperature goes below zero. An overrun-mode for that after key-off is acceptable for a duration of approx. 60 sec. The maximum power-off rate is limited to 46 kW/s due to BOP limitations.

- **Start/stop cycle:** The stack (tested on short stack) shall withstand 150000 start-stop cycles during overall FC lifetime. The number of cycles will be significantly lower for durability testing. The degradation rate shall be recorded and an extrapolation to 150000 cycles can be made.

- **Start-up/shut-down cycle:** The stack (tested on short stack) shall withstand 35000 start-up/shut-down cycles during overall FC lifetime. The number of cycles will be significantly lower for durability testing. The degradation rate shall be recorded and an extrapolation to 35000 cycles can be made.

- **Dynamic drive cycle**: For durability testing the full scale stack shall undergo a dynamic power cycle derived from the NEDC-drive cycle according to the EU harmonization protocols.

The normal operating range for the fuel cell stack is listed in table1:

**Table 1:**
**Operating range of the fuel cell stack**

|  | Peak power | Continuous power |
|---|---|---|
| Cell voltage [V] | 0.6 | 0.659 |
| Current density [A/cm²] | 2.5 | 1.6 |
| Cell temperature [°C] | 85 - 95 | 50 – 85 |
| **Anode** |  |  |
| Stoichiometry (recirculation) | 1.1 – 1.4 | 1.3 – 1.5 |
| Pressure [bara] | 2.2 - 2.5 | 1.5 – 2.2 |
| Min. Rh [%] | 30 | 30 |
| Min. $H_2$-conc. [%] | 40 | 40 |
| **Cathode** |  |  |
| Stoichiometry | 1.8 | 1.8 – 2.0 |
| Pressure [bara] | 2.2 - 2.5 | 1.5 – 2.2 |
| Min. RH / % | Water injection | Water injection |

# 3 DISCUSSION

This chapter describes the actual test sequences for:

1   Leakage test
2   Cell break-in
3   Polarisation curves
4   Freeze start
5   Start/stop procedure
6   Start-up/shut-down procedure
7   Dynamic load cycle

## 3.1   Leakage test

The objective of the leakage test is to prove gas tightness of the fuel cell stack and to measure the leakage rate by pressure decrease in the trapped volume in the cathode and anode side as specified in the requirements specification. The trapped volume includes hoses, manifolds and flow field volume between two valves and outlet ports that enclose the measurement set up.

Procedure:
1.   Flush dry nitrogen flow into the anode and cathode compartment with an open outlet port.
2.   Pressurize the anode & cathode compartment to 3 bar (abs). The pressure difference between anode and cathode during pressurizing must not exceed 0.8 bar.
3.   Reduce nitrogen flow to zero.
4.   Close outlet ports.
5.   Keep cell at this state for 10 minutes.
6.   Measure pressure decrease in both compartments for 20 minutes.
7.   Calculate data to give [mbar * litre * sec$^{-1}$] for the anode and cathode side.

## 3.2   Cell break-in

The following suggestion should be used if specific instructions are not provided by the MEA manufacturer:

1.   Set the cell pressures to 210 kPa (abs) on Anode and 200 kPa (abs) on Cathode (flow rates equivalent for a current density of 0.3 A/cm$^2$).
2.   Increase the current density in steps of 100 mA/cm$^2$ up to 800 mA/cm$^2$ while keeping the cell voltage > 400 mV, and let the cell stabilize for 6 hours. Allow cell temperature to reach above 80°C.
3.   Increase the current while keeping the cell voltage > 400 mV up to 1 A/cm$^2$. Hold the cell in these conditions for at least 2 hours.
4.   Stability validation by 0.4 V to 0.8 V cycles with stoichiometric condition.
5.   At the 1.0 A/cm$^2$ set point perform the reference 1 recording for 5 min, move to a high potential (0.8 V) and hold for 5 min. Subsequently move to a low potential (0.4 V) and hold for 5 min also. Repeat these jumps till three high voltage and two low voltage plateau measurements have been performed and hold for 0.5 h at the 1.0 A/cm$^2$ set point to record the reference 2 for 5 min.

The recommended stability criterion is based on the cell voltage fluctuation and it is considered fulfilled when it is lower than ±5 mV in the reference 2 step. Additionally, the cell voltage fluctuation between the

reference time frame 1 and 2 should be lower than ±10 mV. For higher fluctuation the whole or parts of the procedure have to be repeated till the targets are reached.

## 3.3 Polarisation curves

The objective of performing polarisation (IV curve) measurements is to determine the stack performance in terms of cell voltage and power density against current density at specified operating conditions.

A conditioning step is required before the actual test starts. Cell conditioning starts by setting the required operating parameters to the reference or the stressor conditions (table 3, @1 A/cm²) and maintain these conditions until the cell voltage is stable that is when it varies by not more than +/-5 mV during the last hour of conditioning procedure. In case no cell stability is obtained within 6 hours of operation a repetition of the break-in and conditioning should be reconsidered.

The dwell time of each set point should be long enough to ensure that stabilisation criteria of cell voltage of ±5 mV over 2 min (<15 min) except for the OCV which shall not exceed 1 min dwell time. The proposed set points are summarised in Table 1.

High/low stressors conditions (table 3) will be applied to the polarization curve to determine influence on the stack power density.

**Table 1 -** Polarisation curve set-points

| Set point no. | Current density A/cm² | 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 | 1.00 | ≥120 | ≥30 |
| 12 | 1.40 | ≥120 | ≥30 |
| 13 | 1.80 | ≥120 | ≥30 |
| 14 | 2.00 | ≥120 | ≥30 |
| 15 | 2.20 | ≥120 | ≥30 |
| 16 | 2.40 | ≥60 | ≥30 |
| 17 | 2.50 | ≥30 | ≥30 |
| 18 | 2.40 | ≥60 | ≥30 |

**Table 1** – continued: **Polarisation curve set-points**

| Set point no. | Current density A/cm² | Recommended dwell time (s) | Recommended Data acquisition time (s) |
|---|---|---|---|

| 19 | 2.20 | ≥120 | ≥30 |
|----|------|------|-----|
| 20 | 2.00 | ≥120 | ≥30 |
| 21 | 1.80 | ≥120 | ≥30 |
| 22 | 1.40 | ≥120 | ≥30 |
| 23 | 1.00 | ≥120 | ≥30 |
| 24 | 0.60 | ≥120 | ≥30 |
| 25 | 0.40 | ≥120 | ≥30 |
| 26 | 0.30 | ≥120 | ≥30 |
| 27 | 0.20 | ≥120 | ≥30 |
| 28 | 0.10 | ≤60 | ≥30 |
| 29 | 0.08 | ≤60 | ≥30 |
| 30 | 0.06 | ≤60 | ≥30 |
| 31 | 0.04 | ≤60 | ≥30 |
| 32 | 0.02 | ≤60 | ≥30 |
| 33 | 0.00 | ≤60 | ≥30 |

If the maximum current density of 2.5 A/cm$^2$ cannot be reached, the end point of the polarisation curve will be at the closest current setting giving a minimum cell voltage of 0.4 V. If higher current density settings than 2.5 A/cm$^2$ are possible, it is recommended to continue recording in 0.1 A/cm² steps until a cell voltage of 0.4 V is obtained.

## 3.4  Freeze start

Freeze start starts at a stack temperature below 0 °C. The specific requirement was set at -25 °C. The gases do not need to be conditioned to -25 °C. The stack can be heated up to a temperature high enough to allow for a stable operation (e.g. 0 °C), either by a specific power/efficiency strategy or using an external heating device. The total energy consumption must not exceed a specified amount of energy. The time necessary to reach 50 % of the nominal power (90 kW) after "key-on" shall be recorded. Each freeze start contributes to a portion of the overall stack degradation which limits the remaining life time.

The freeze start up endurance test starts with the appropriate cell leak test, break-in and conditioning as described in 3.2 and 3.3 and is performed according to the following protocol:

1. Set the test to standard operating conditions.
2. At beginning of test (BoT) and periodically through the test, perform a polarization curve measurement or record cell voltage at 0.1; 0.8 and 1.8 Acm$^{-2}$. Record also the average current density at 0.675 V (as average of ascending and descending polarisation curve measurements). This value of current is used as 100% current density. 50% of this value is the set point for the freeze start procedure. An EIS sweep is also recorded.
3. Stack shut down
   a. Set current to 0 A.
   b. Stop reactant supply (Air & H$_2$) and water cooling at the cell inlet and depressurise the anode compartment.
   c. Allow cathode to blow-down (constant flow of air for 5 min to remove excess water from the stack & clear water lines).
   d. Leave to cool to -25 $^0$C over 6 hr.
4. Stack Start up

a. Open $H_2$ inlet & outlet valves to enable fast purge of anode compartment with $H_2$.
b. Set air flow rates for initial base load operating conditions (0.2 A/cm$^2$ at 8 air stoichiometry.
c. Engage contactor
d. Modulate current up to base load on the following;-
    i. Increase current if cell voltage > 0.35V.
    ii. Decrease current if cell voltage <0.35V.
e. Once a specified amount of electrical energy has been generated (& released liquid water), pump coolant water into stack at operational flow rates.
f. Increase the current (according to the rules specified in 4d) to 50% nominal power.
g. Operate the cell at the specified operating conditions for 10 minutes and record the cell voltage while averaging the last 60 seconds of recording.

5. Repeat steps 3 to 7, 20x. Re-benchmark stack performance by repeating steps 1 & 2 [approx. weekly]. The test ends (EoT) after 40 freeze starts, or a degradation rate has been established (with < 10% error of the slope).
6. At (EoT), perform measurements as in step 2.

## 3.5   Start-stop procedure

The EU harmonization protocol utilises the ON-OFF Cycling Endurance test to stress the fuel cell by means of instantaneous and alternating phases of on and off loads causing frequent changes in pressure and temperature which are more abrupt for pressure changes rather than temperature changes. Modifications of this test are required for IE evaporatively-cooled hardware. This test maintains a $H_2$ atmosphere on the anode.  The cell is tested by applying loads at nominal continuous power (ON phase) followed by a period of shutdown (OFF phase) where the load (current) is set to zero. The cell voltage is recorded during the ON phase to observe its evolution. At BoT and EoT, polarisation curve measurements and additional diagnostic testing including cyclic voltammetry, fuel crossover and EIS measurements are performed.

The start-stop Cycling Endurance test starts with the appropriate cell leak test, break-in and conditioning as described in 3.2 and 3.3 and is performed according to the following protocol:

1. Set the test to standard operating conditions.
2. At BoT & periodically through the test, perform a polarization curve measurement or record cell voltage at 0.1; 0.8 and 1.8 A/cm$^2$. Record also the average current density at 0.675 V (as average of ascending and descending polarisation curve measurements). This value of current is used as 100% current density. An EIS sweep is also recorded.
3. Increase the current density to 100%.
4. Operate the cell at the specified operating conditions for 2 minutes and record the cell voltage while averaging the last 30 seconds of recording.
5. Decrease the current load to zero current.
6. Stop reactant supply (Air & $H_2$) at the cell inlet (no purge). The anode pressure will be reduced over time due to internal & external leaks.
7. Wait for 1 min.
8. Restart the cell by setting the initial operating conditions and have the cell voltage stabilised.
9. Repeat steps 3 to 8, 3000x. Re-benchmark stack performance by repeating steps 1 & 2 [approx. weekly]. The test ends (EoT) after 10,000 start/stop cycles, or a degradation rate has been established (with < 10% error of the slope).
10. At (EoT), perform measurements as in step 2.

## 3.6    Start-up/shut-down procedure

The stack is tested by applying loads at nominal continuous power (ON phase) followed by a period of shutdown (OFF phase) where the load (current) is set to zero. The cell voltage is recorded during the ON phase to observe its evolution. At BoT, EoT & periodically through the test cycles, polarisation curve measurements and additional diagnostic testing including cyclic voltammetry, fuel crossover and EIS measurements are performed.

The start-up/shut-down cycling endurance test starts with the appropriate cell leak test, break-in and conditioning as described in 3.2 and 3.3 and is performed according to the following protocol:

1. Set the test to standard operating conditions.
2. At BoT & periodically through the test, perform a polarization curve measurement or record cell voltage at 0.1; 0.8 and 1.8 Acm$^2$. Record also the average current density at 0.675 V (as average of ascending and descending polarisation curve measurements). This value of current is used as 100% current density. An EIS sweep is also recorded.
3. Increase the current density to 100%.
4. Operate the cell at the specified operating conditions for 10 minutes and record the cell voltage while averaging the last 60 seconds of recording.
5. Stack shut down
   a. Set current to 0 A.
   b. Stop reactant supply (Air & $H_2$) & water cooling at the cell inlet & depressurise the anode compartment.
   c. Blow down cathode compartment to remove excess water
   d. Leave to cool over 20 minutes & the cell voltage to depolarise
   e. Purge the anode compartment with Air.
6. Stack Start up
   a. Open $H_2$ inlet & outlet valves to enable fast purge of anode compartment with $H_2$.
   b. Set air & water flow rates for initial operating conditions.
   c. Engage contactor & draw appropriate load.
7. Repeat steps 3 to 7, 300x. Re-benchmark stack performance by repeating steps 1 & 2 [approx. weekly]. The test ends (EoT) after 1,000 start/stop cycles, or a degradation rate has been established (with < 10% error of the slope).
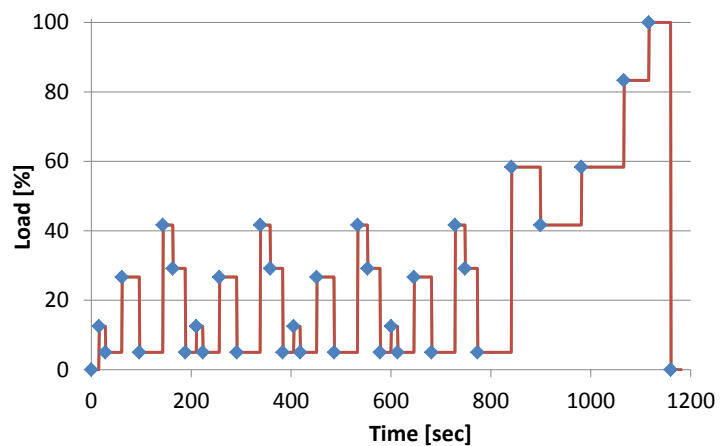8. At (EoT), perform measurements as in step 2.

## 3.7    Dynamic load cycle

The proposed Fuel Cell Dynamic Load Cycle (FC-DLC) is based on the EU-harmonization test protocol and is defined by 35 Test Points (TP) or steps as described in table 2 and shown in figure 1. The test duration of one complete cycle is 1181 seconds. A total of 1400 cycles shall be performed which corresponds to approximately one year of vehicle operation (or 15400 km driving).

**Table 2 -** FC-DLC test points time/load                    **Figure 1**: Normalized load cycle

| Step | Time [sec] | Dwell [sec] | Load [%] |
|------|-----------|-------------|----------|
| 1 | 0 | 15 | 0.0 |
| 2 | 15 | 13 | 12.5 |
| 3 | 28 | 33 | 5.0 |
| 4 | 61 | 35 | 26.7 |
| 5 | 96 | 47 | 5.0 |
| 6 | 143 | 20 | 41.7 |
| 7 | 163 | 25 | 29.2 |
| 8 | 188 | 22 | 5.0 |
| 9 | 210 | 13 | 12.5 |
| 10 | 223 | 33 | 5.0 |
| 11 | 256 | 35 | 26.7 |
| 12 | 291 | 47 | 5.0 |
| 13 | 338 | 20 | 41.7 |
| 14 | 358 | 25 | 29.2 |
| 15 | 383 | 22 | 5.0 |
| 16 | 405 | 13 | 12.5 |
| 17 | 418 | 33 | 5.0 |
| 18 | 451 | 35 | 26.7 |
| 19 | 486 | 47 | 5.0 |
| 20 | 533 | 20 | 41.7 |
| 21 | 553 | 25 | 29.2 |
| 22 | 578 | 22 | 5.0 |
| 23 | 600 | 13 | 12.5 |
| 24 | 613 | 33 | 5.0 |
| 25 | 646 | 35 | 26.7 |
| 26 | 681 | 47 | 5.0 |
| 27 | 728 | 20 | 41.7 |
| 28 | 748 | 25 | 29.2 |
| 29 | 773 | 68 | 5.0 |
| 30 | 841 | 58 | 58.3 |
| 31 | 899 | 82 | 41.7 |
| 32 | 981 | 85 | 58.3 |
| 33 | 1066 | 50 | 83.3 |
| 34 | 1116 | 44 | 100.0 |
| 35 | 1160 | 21 | 0.0 |



The load cycling starts with the appropriate cell leak test, break-in and conditioning as described in 3.2 and 3.3 and is performed according to the following table 2. In this specific case, a power demand of 100 % is defined as the power generated at 0.659 V at BoL (approximately 90 kW).

1. Set the test operating conditions at the reference conditions, table 3.
2. At the Beginning of Test (BoT), perform a polarization curve according to 3.3 or record the cell voltage at 0.1, 0.8 and 1.8 A/cm$^2$ with dwell times as specified.

3. Operate the cell at the FC-DLC driving cycle for 100 consecutive cycles (100 cycles = one test block).

4. At the end of 100 FC-DLC perform a polarization curve or record the cell voltage at 0.1; 0.8 and 1.8 A/cm$^2$ and compare with measurements performed at BoT. Observe the degradation rate in µV/hour.

5. Repeat steps from 3 to 4 up to a total of 1400 driving cycles.

**TABLE 3**
**Low and High Values of Stressor Conditions**

| | Parameters | Symbol | Unit | Operating conditions | | |
|---|---|---|---|---|---|---|
| | | | | Low Settings (L) | Reference Settings (R) | High Settings (H) |
| | Nominal cell operating temperature | $T_{cell}$ | °C | Result of EC cooling and lambda air | Result of EC cooling and lambda air | Result of EC cooling and lambda air |
| **ANODE** | Fuel gas inlet temperature single cell only | $T_{fuel,in}$ | °C | 50 | 85 | 100 |
| | Fuel gas inlet humidity | $RH_{fuel,in}$ | % | recirculation | recirculation | recirculation |
| | Fuel gas inlet pressure (abs.) | $p_{fuel,in}$ | kPa | 160 | 250 | 300 |
| | Fuel gas composition | $x_{H2, fuel}$ | - | $H_2$ 5.0 quality (<0.5 ppm CO, <0.5 % $N_2$, <0.5 ppm $CO_2$) | | |
| | Fuel inlet stoichiometry | $\lambda_{H2}$ | - | 1.1 | 1.3 | 1.5 |
| **CATHODE** | Oxidant gas inlet temperature | $T_{air,in}$ | °C | 50 | 85 | 100 |
| | Oxidant gas inlet humidity, single cell only | $RH_{Ox,in}$ | % | 20 | 30 | 85 |
| | Oxidant inlet pressure (abs.) | $p_{Ox,in}$ | kPa | 140 | 230 | 280 |
| | Oxidant gas composition | $x_{Air, Ox}$ | - | Air (<500ppm $CO_2$, ≤10ppm CO, ≤2.5 ppm NO+$NO_2$, ≤5ppm $SO_2$, ≤0.01 mg/m$^3$ Oil) | | |
| | Oxidant inlet stoichiometry | $\lambda_{Ox}$ | - | 1.3 | 1.5 | 2.0 |

# 4 CONCLUSIONS AND FUTURE WORK

Test requirements and high level test procedures have been described. Test results of short stack and full scale stack tests will be compared to the requirements specification and the corresponding pass/fail

criteria. If appropriate and in agreement with all partners, the described procedures may be adjusted in order to accelerate degradation effects and fine tune testing time.

# 5   REFERENCES

EU Harmonization Test Protocol:
Georgios Tsotridis, Alberto Pilenga, Giancarlo De Marco, Thomas Malkow; EU HARMONISED TEST PROTOCOLS FOR PEMFC MEA TESTING IN SINGLE CELL CONFIGURATION FOR AUTOMOTIVE APPLICATIONS; JRC Science for Policy report, 2015; EUR 27632 EN; doi 10.2790/54653