Test report

Test number	TV501006_2
Test descriptive name	H2Fly High Temperature (HT) DOE test

Abstract

Topic:
High temperature (HT) design-of-experiment (DOE) test on a P10 short-stack integrated with a custom HT MEA.

Target:
To construct and perform a DOE which maps the P10 stack (with HT MEA) behavior within the high temperature and high current regions of operating conditions relevant to H2Fly.

Test info table

Test responsible	Abdinasir Farhan
Test object	23-cell P10-stack with custom HT MEA
Test station	*Greenlight Innovation G400*
Test period	July-Sep 2024
Test number	TV501006_2

Conclusions

- 600 DOE test points were planned, of which 560 TP were successfully completed (corresponding to a 93% completion). This makes the P10 stack integrated with the custom HT MEA a suitable stack platform for the temperature and current ranges that H2Fly requests.
- The vast majority uncompleted test points were due to mass transport losses related to high currents (>600A) in combination with low reactant inlet pressures and/or stoichiometries. Two data points could not be completed due to test station limitations.
- Reference polarization curves show performance loss in the kinetic region (<150A) believed to be caused by loss of catalyst related to the severity of the DOE test.
- Comparing the ohmic and mass transport regions of the polcurves, performance is nearly identical which indicates that the membrane is virtually pristine after more than 200 hours of testing, including high temperature testing.

Future work and recommendations

- Load the generated test data into a statistical model, for the custom P10-stack, which can establish the best operating conditions that yield optimal output performance in the DOE range. Furthermore, the effects of individual parameters on stack performance can be isolated and finetuned.

- Compare the results from the present design of experiment test with the DOE performed previously on the standard P10-stack for H2Fly. By loading the two datasets into data driven models, the differences between two MEA types can be highlighted at given operating conditions. Furthermore, the statistical models would work as a complement to physical model for the P10-stack.

- Possible follow-up test to study mass transport losses and their causes as well as possible strategies to mitigate these losses. This is especially suitable if H2Fly desires to operate the stack at high currents since mass transport losses are highly significant at these loads.

Revision log

Revision	Date	Change	By
a0	2024-09-20	First version	Abdinasir Farhan

Nomenclature

BoL	*Beginning of life*
EoL	*End of life*
BoT	*Beginning of life*
EoT	*End of Life*
CV	*Cyclic voltammetry*
EIS	*Electrochemical impedance spectroscopy*
DOE	*Design of Experiment test*
MEA	*Membrane electrode assembly*
Delta T	*Difference between coolant inlet vs outlet temperature*

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1 Introduction

H2Fly are interested in applications that push the upper limit of operating temperatures for the P10 stack platform. At such elevated temperatures, the standard P10 MEA is not optimal. Thus, an alternative, high temperature (HT) MEA integrated in the P10 stack has been suggested. The high temperature MEA is of course excellent in normal operating conditions as well.

Due to the multivariate nature of fuel cell operation, a design of experiment (DOE) test was considered as the best methodology for accurate characterization of the HT MEA. This methodology allows for the organization and testing of the numerous operating conditions that can be used to map a fuel cell operating window. Most recently, DOE methodology was used for a standard P10 stack for H2Fly. Within this follow-up test, the boundary conditions for the stack outlet temperature are increased. Specifically, stack outlet temperatures of up to 105°C are targeted.

D-optimal Latin hypercube sampling was the statistical method used to generate DOE data points based on boundary conditions set for each input parameter. The input parameters include stack coolant outlet temperature, difference between coolant inlet and outlet temperature (dT), coolant pressure, delta P (pressure differential between cathode pressure and anode pressure), anode/cathode stoichiometries and inlet pressures, hydrogen concentration and current. The mean cell voltage was chosen as the response parameter with a final sample size of 600 points.

1.1 Purpose

The main purpose of this test is to, via DOE methodology, map performance of the HT MEA in the high temperature and high current window.

1.2 Scope

This test is the same size (600 sample points) as the previous DOE performed for H2Fly on the standard P10 stack. The main difference is the higher upper boundary for the stack outlet temperature (105 °C) and a different current range (400-800A). Another difference is the use of a short stack. However, a 23-cell short stack can be considered a representation of the behavior of a full stack.

The test object is a 23-cell P10 stack with a custom, high temperature MEA. As a complement to the DOE, a reference polarization curve was planned to be performed at BoT and EoT to study the effects of the design of experiment test. The stack was at the beginning of life at the start of the test.

2 Method

2.1 Test object and equipment

Stack was activated on 2024-06-14 followed by BoT polarization curve. The stack was then on shelf until 2024-07-15 due to a test station uppgrade.

Stack type	P10 (custom MEA)
Number of cells	23
Activation date	2024-06-14

2.2 Test procedure

The test campaign was planned to run in the following order:

1) Beginning of test (BoT) polarization curve for reference, including conditioning.
2) Design of experiment test, spread across 600 test points.
3) End of test (EoT) polarization curve for reference including conditioning.

During the DOE test, a further polarization curve was added to the test campaign and performed after ca 480 test points: just before the high temperature part of the DOE. This will yield information about the effects of high temperature operation. The operating conditions for the reference polcurve are presented in Appendix. The complete test plan can be found in reference [1].

As mentioned in section 1, the DOE OpCons were generated using D-optimal Latin Hypercube sampling with the following input variables:

- General: current, Anode-cathode pressure diff.
- Anode: inlet pressure, inlet relative humidity, stoichiometry, H_2 concentration.
- Cathode: inlet relative humidity, stoichiometry.
- Coolant: delta T, outlet temperature.

Boundary conditions for the DOE input variables above were set with regards to previously known P10-stack operating range, test station specifications and costumer requirements. The most noteworthy input variables for the customer are current and coolant outlet temperature. Remaining operating parameters such as coolant inlet temperature and gas temperatures were set with respect to the input variables above. The list of all input variables for the two temperature test windows is found in reference [2].

The step times reported in presented in table 1 were an estimate and depended on the feasibility of the generated DOE test points. The test points were ordered with respect to coolant inlet temperature and were run in chronological order from lowest temperature to highest temperature. Each test point was held for 15 minutes after transition, excluding wait times to reach temperature stabilization. Transition times were different for each test point and depended mainly on current ramping and temperature changes.

The average cell voltage was set as the response/output parameter for the DOE. Test points which yielded a minimum cell voltage below 0.3V were stopped by test station and deemed unfeasible to run. The minimum cell voltage was decreased from 0.3V to 0.25V during the last 70 test points. This was done to reduce the number of stops which disrupted the test since it took a long time to restart at the next test point.

Table 1. Test procedure.

Step	Type of protocol	Protocol/script/SOC reference	Step time (h)
1	Conditioning		2
2	Polarization curve BOT		3
3	DOE test	2_Full Test window - H2Fly High Temperature DOE - 2024-06-01_CC.xlsx	220
4	Conditioning		2
5	Polarization curve EOT		3
			Total:230

2.3 Data analysis and storage

As mentioned in section 2.2, all test points were allowed to run for 15 minutes after transition to reach steady state. All input and output parameters measurements were then averaged across 50 seconds at steady state. These averaged values are presented in this document [3].

3 Results & discussion

The result from the DOE test is presented in this section. Furthermore, reference polarization curves are presented. Supporting material for the polcurve is found in the Appendix. The test was executed generally as planned in the test plan with some deviations:

1) 40 test points (ca 7% of all test points) could be not performed due to either mass transport losses or test station limitations.

2) The minimum allowable cell voltage was decreased after test point 523 from 0.3V to 0.25V. The decrease was implemented to decrease the amount of emergency stops and to better map the higher current region.

3) A third polarization curve was performed after 480 test points (just before high temperature region). This allowed for isolation of the effects of hot operation on the MEA compared to the rest of the DOE.

3.1 Performance

Figure 1 presents the reference polarization curves taken before, and after the DOE test. Furthermore, the third polarization curve performed just before the high temperature region of the DOE is also seen in Figure 1. The polcurves were performed to verify that the quality of the DOE results is not affected by degradation.

Comparison of the polcurves shows decreased performance in the activation polarization region (<150A) caused by loss of catalyst. This loss of catalyst is mainly due to the natural severity of the DOE test during which many unusual operating conditions are tested to map the operating limits of the stack platform.

For load currents above 150A, it seems that the performance difference between the BoT and EoT is smaller. This is possibly due to a more humidified membrane at EoT leading higher proton conductivity and reduced ohmic losses which offsets the loss of catalyst at higher currents. The identical performance in the ohmic region also suggests that the membrane is still pristine even after high temperature operation.

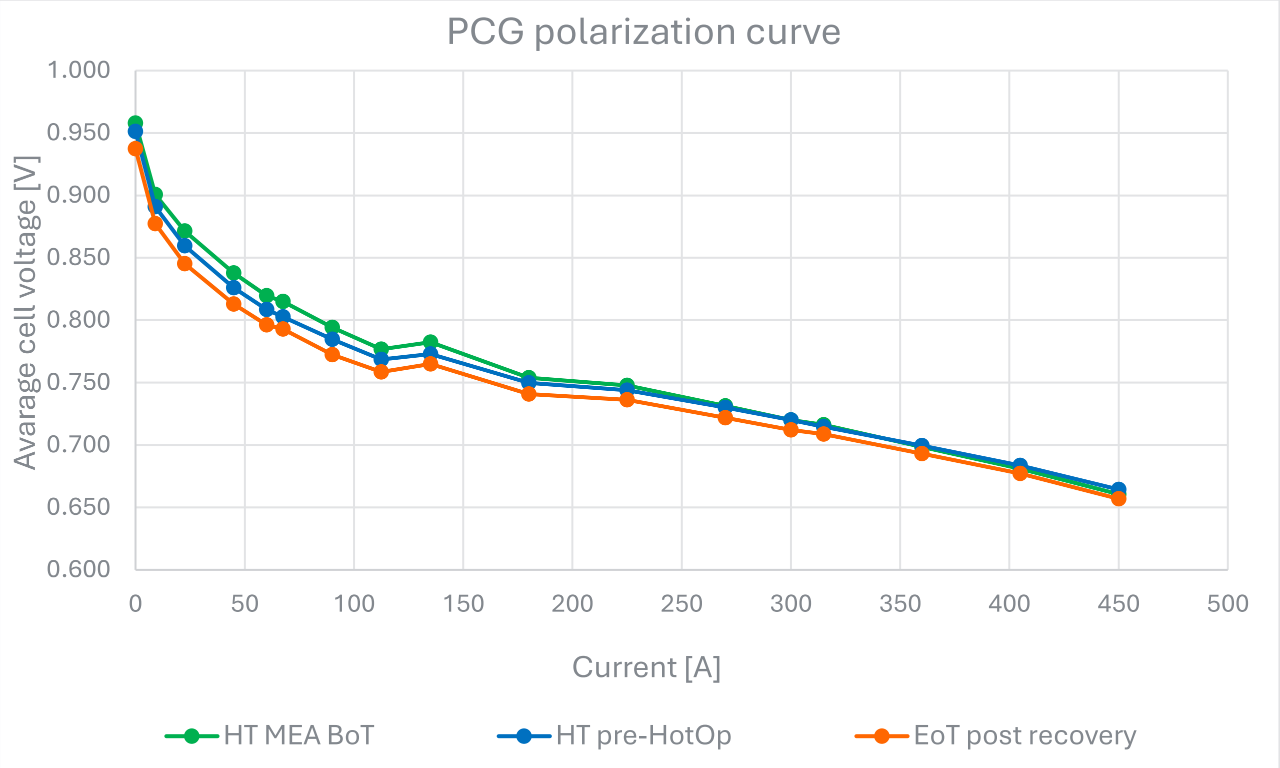

Figure 1 Reference polarization curves taken at BoT, before hot operation (HotOp) and at EoT.

3.2 DOE

As presented in the test procedure, 600 test points were planned for this test campaign. Test points which yielded a minimum output cell voltage of 0.3V or lower were discarded. For the last 77 test points the minimum allowable cell voltage was reduced 0.25V. A total of 560 test points were completed which gives >93% completion rate for the DOE. Almost all the remaining 40 test points that could not be completed were due to combinations of high current and low inlet pressures and/or low stoich leading to large mass transport losses.

Some of the completed data points had to be restarted due to test station constraints or script logic reasons. These include too fast flow changes or pressure increases. Control system behavior for the coolant loop was another reason for restarts. During transitions between test points, almost all parameters are changed at the same time, and occasionally, the generated heat from fuel cell operation caused the test station control system to become unstable and cause the coolant temperature to deviate from setpoint. Restarting an affected test point from room temperature often fixed this issue.

Due to the large number of test parameters and test points, presenting the DOE in this report is difficult. The averaged values for input and output parameters for all 560 completed test points are instead presented in this document [2]. Figures 2 and 3, illustrate completed load current and stack temperature for each test point in relation to the corresponding target values.

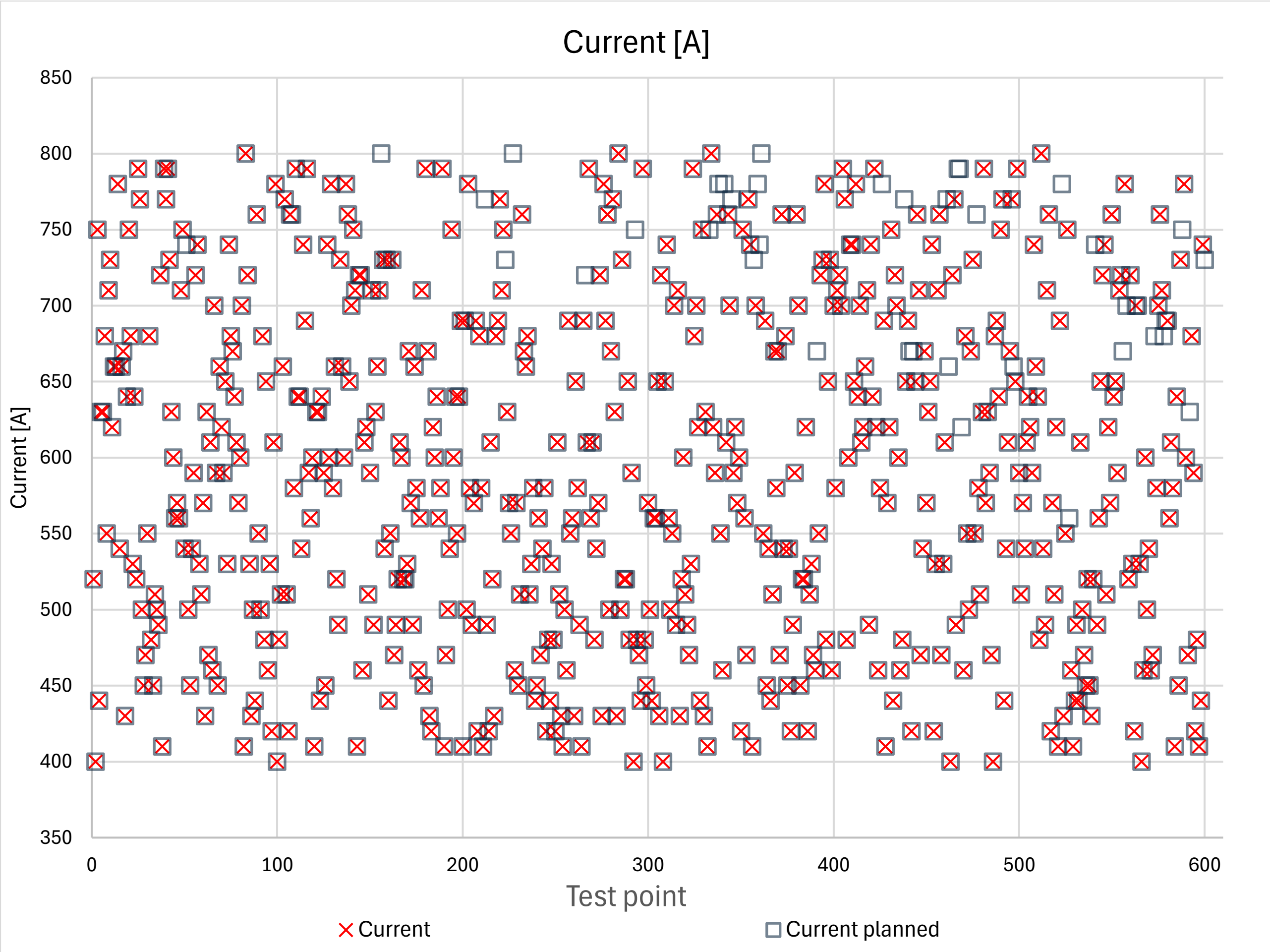

Figure 2. Planned vs performed test points. Data points are missing mainly at high currents due to large mass transport losses which lead to low output voltages.

Figure 2 shows that nearly all skipped test points are at currents above 600A. As mentioned before, when these high currents are coupled with low stoichiometries or pressures, mass transport losses increase drastically since reactant gases are consumed faster than they are supplied.

The effect of the mass transport losses is that at high enough load currents the voltage reaches a cut-off point and crashes. The test station turns off the test if the cell voltage reaches the minimum allowable value. This was done for the safety of the stack, which is also the reason for the uncompleted tests points. Observations showed that cathode pressure and stoich has a much larger effect on mass transport losses than the anode pressure and stoich.

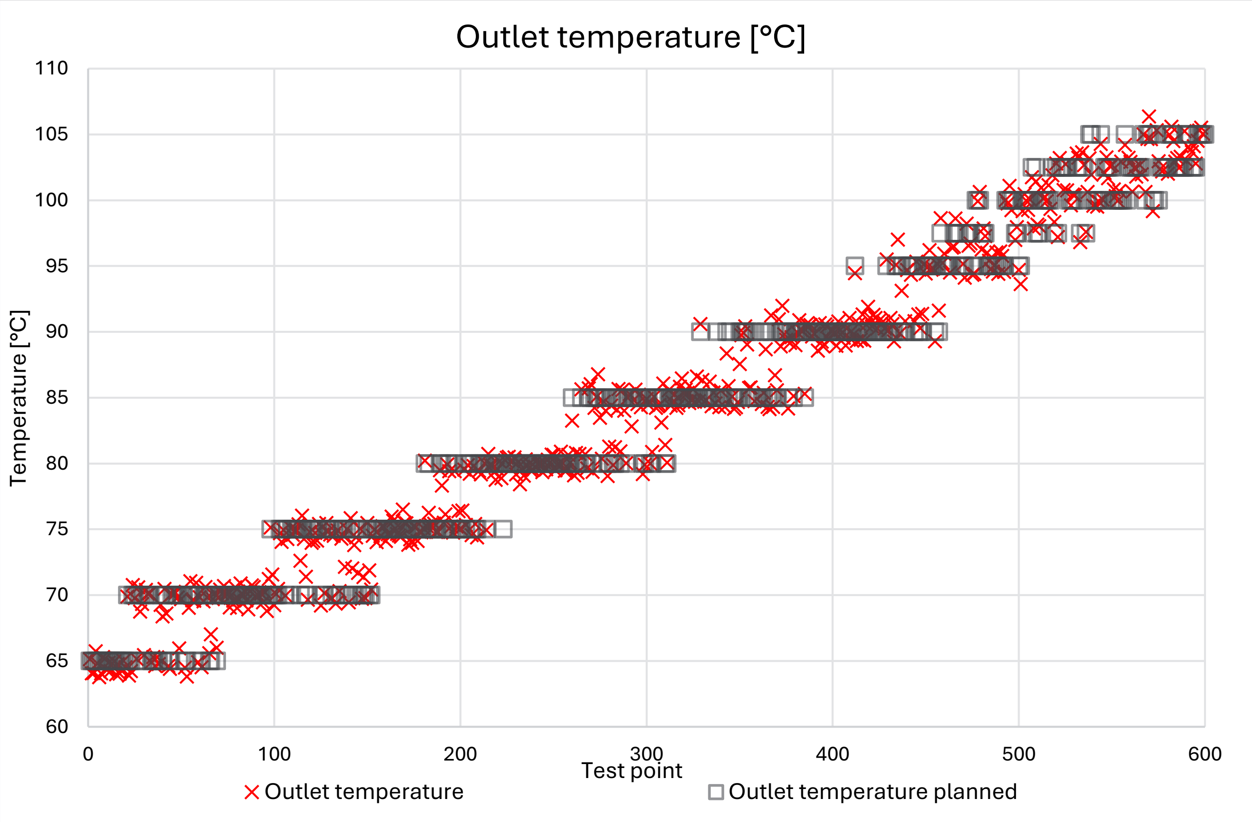

Figure 3 Stack outlet temperature vs targeted outlet temperature. Spread due to variation in generated heat.

Figure 3 above shows the same 600 test points distributed across the target temperature range. During the first 480 test points the target temperature is increased in 5°C increments. Above 95°C, the resolution is changed to 2.5°C [2]. The spread in temperature is within ±2°C of setpoint and is caused by test station coolant control system. The large amounts of heat generated at high currents, along with the rapid transitions of all operating conditions make holding the setpoint temperature difficult for the test station controllers. The spread in temperature was largely reduced, via PID-tuning, in the latter stages of the DOE.

4 Conclusion

- 600 DOE test points were planned, of which 560 TP were successfully completed (corresponding to a 93% completion). This makes the P10 stack integrated with the state-of-the-art MEA a highly suitable platform for the temperature and current ranges that H2Fly requests.

- The vast majority uncompleted test points were due to mass transport losses related to high currents (>600A) in combination with low reactant inlet pressures and/or stoichiometries. Two data points could not be completed due to test station limitations.

- Reference polarization curves show performance loss in the kinetic region (<150A) believed to be caused by loss of catalyst related to the severity of the DOE.

- Comparing the ohmic and mass transport regions of the polcurves, performance is nearly identical which indicates that the membrane is virtually pristine after more than 200 hours of testing, including high temperature testing.

4.1 Fulfilling of hypothesis

Due to the multivariate nature of the DOE test, most of the conclusions below are general observations made while running the test object.

- The stack and high temperature MEA performed adequately in the temperature range below 95 °C as expected. This was confirmed by polarization curves at standard operating temperature as well as the general output voltage from DOE test points. Furthermore, most of the high temperature test points were successfully completed and need to be further validated.

- The custom P10-stack performed well at currents up to 600A at nearly all combinations of operating parameters within the boundary conditions of the DOE. Additionally, the stack performed adequately at currents above 600A if coupled with high enough reactant gas inlet pressure and stoich.

- The HT MEA was able to operate at temperatures up to 105°C without any noticeable degradation to membrane performance. Polarization curve shows some loss of catalyst after completed DOE believed to be caused by the harsh operating conditions of the DOE.

4.2 Recommendations

The following list contains recommendations on how to proceed after this test

- Load generated test data into a statistical model, for the custom P10-stack, which can establish the best operating conditions that yield optimal output performance in the DOE range. Furthermore, the effects of individual parameters on stack performance can be isolated.

- Compare the results from the present design of experiment test with the DOE test performed previously on the standard P10-stack for H2Fly. By loading in the two datasets into data driven models, the differences between two MEA types can be highlighted at given operating conditions. Furthermore, the statistical models would work as a complement to the physical model for the P10-stack.

- Possible follow-up test to study mass transport losses and their causes as well as possible strategies to mitigate these losses. This is especially suitable if H2Fly desires to operate the stack at high currents since mass transport losses are highly significant at these loads.

5 References

[1] Abdinasir Farhan (2024), Test plan: H2Fly High Temperature DOE. [2024-06-15] URL: 1_Test plan - H2Fly High Temperature DOE - 20240527_CC.docx

[2] Abdinasir Farhan (2024), Full Test window - H2Fly High Temperature DOE. [2024-09- 16] URL: 2_Full Test window - H2Fly High Temperature DOE - 2024-06-01_CC.xlsx

[3] Abdinasir Farhan (2024), H2FLY HT DOE - Averaged Test Point values - [2024-09-18]. URL: H2FLY HT DOE - Averaged test points - 20240912_CC.xlsx

Appendix

Table 2 below describes the operating conditions for the reference polarization curve performed during the test campaign.

Table 2 Operating conditions used in the reference polarization curve.

Current [A]	P_air_inlet [barg]	P_H2_inlet [barg]	Stoic_air	Stoic_H2	T_coolant_in [℃]	Flow_coolant [LPM]/cell	T_gas_in [℃]	RH air [%] **	DPT_air [°C]	DPT_H2 [°C]	RH fuel [%] **	Fuel H2_ conc_inlet [%]
0	0.2	0.4	***	***	65	0.45	70	55.8	52.5	52.5	55.8	70
9			36	30								
22.5			14.4	12								
45			7.2	6								
60			5.4	4.5								
90	0.3	0.5	3.6	3								
112.5			2.9	2.4								
135	0.7	0.9	2.4	2								
180			1.8	1.5								
225	1	1.2	1.8	1.5								
270												
300												
315												
360												
405												
450												

** Calculated with reference to coolant inlet temperature
*** Same flows as previous load point
0-180A flows are kept at the flows corresponding to 180A