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Connectors tests under high pressure hydrogen

[Tests de connecteurs sous haute pression d'hydrogène]

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Connectors tests under high pressure hydrogen

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Résumé

Français

Ce rapport présente les tests sous haute pression d'hydrogène réalisés au centre CEA-Valduc dans le cadre du projet européen EIHP2.

Les objectifs de ce projet sont de définir un système de remplissage des réservoirs des futurs véhicules à hydrogène mais également de contribuer à la définition d'une réglementation européenne pour ce nouveau moyen de transport.

Dans une première phase deux connecteurs conçus pour le gaz naturel par STAUBLI ont été testés sous hydrogène pour la détection des fuites et le comportement thermique au court d'une détente d'hydrogène à l'atmosphère.

Dans une seconde phase les connecteurs ont été interfacés à un réservoir de 9 litres pour simuler le remplissage d'un réservoir de véhicule. Deux autres connecteurs spécialement conçus par STAUBLI pour l'hydrogène (conformément au projet de norme SAE J2600) ont également été testés dans ces conditions.

Anglais

This report presents tests under very high pressure Hydrogen performed at CEA – Valduc in the frame of the European project EIHP2.

The aim of this project is to design a refuelling system for future hydrogen powered vehicles and also to participate in the definition of European regulation governing this new means of transport.

In a first stage two connectors made by STAUBLI for natural gas were tested for leakage detection and thermal behaviour during hydrogen pressure release to atmosphere.

In a second stage the connectors were interfaced with a 9 litres tank simulating the vehicle refuelling. Two other connectors specially designed by STAUBLI for hydrogen (in agreement with SAE J2600 project of standard) were also tested in the same conditions.

Mots clés

Hydrogène, haute pression, test, connecteur, réservoir.

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1 INTRODUCTION

The 3-year European Project, EIHP-2 (European Integrated Hydrogen Project phase 2), that began in 2001 falls within the framework of research into new energy solutions. The aims are to design a refuelling system for future hydrogen powered vehicles and also to participate in the definition of European regulations governing this new means of transport.

This programme unites over 20 partners, including the CEA. The Valduc centre is responsible for experiments on high pressure hydrogen connectors.

Tests were performed on four connectors manufactured by STÄUBLI. Two were connectors marketed for refuelling vehicles running on natural gas, this type of connector – referred to in this study as GN-R&D-01 and GN-R&D-02 (manufacturer references: Test H2-R&D-0001/02 and Test H2-R&D-0002/02) – has already been tested for this type of application up to a pressure of 200 bar (Figure 1). The other 2 connectors were especially developed by STÄUBLI for hydrogen refuelling, references H2-R&D-01 and H2-R&D-02 (manufacturer references: R&D2-03/03 N00641803A 2 and R&D2-03/03 N00751103A 2).



Figure 1. STÄUBLI connector.

The purpose of this report is to present the results of experiments obtained for these connectors by the teams working with the HDT service at the Valduc centre.

Experiments were carried out in two stages. Firstly, connectors GN-R&D-01 and GN-R&D-02 were tested under high pressure Hydrogen with venting. Subsequently, the four connectors were interfaced with a 9 litre tank simulating the vehicle fuel tank.

Operating conditions and results obtained for each of these two stages are presented in the following paragraphs.

2 FIRST TEST PHASE

2.1 Test means

A hydrogen storage system comprising five 10-litre containers and a dedicated high flow-rate hydrogen supply system were designed to perform the first test phase. Experiments initially involved placing the 5 containers under 400 bar pressure and then relieving hydrogen pressure in the connector by opening a pneumatic valve (Figure 2). After passing through the connector, the hydrogen was then expelled outside the building. Installation lines and piping were sized such that they did not limit transfer. The connectors were placed within a confinement tank under rough vacuum in order to measure any leaks by means of an INFICON leak detector (UL 200 dry) and guarantee safety in the event of bursting.

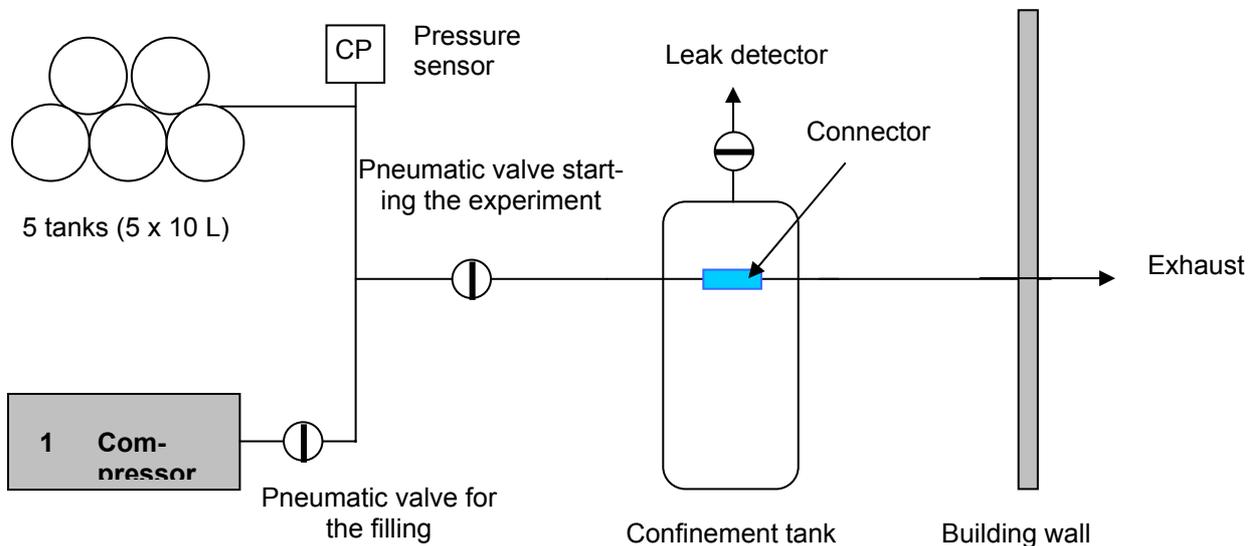


Figure 2. Experimental apparatus used for the first test phase.

Only the GN-R&D-01 and GN-R&D-02 connectors were examined during this first phase (Figure 3).



Figure 3. GN-R&D-01 and GN-R&D-02 connectors.

Pressure variations as well as temperature variations on the external surface upstream and downstream of the connectors (Figure 4) were recorded during testing.



Figure 4. Fitting of thermocouples to the connector.

2.2 Experimental results

The results of this first test phase were presented in a report published in May 2003 (CEA/VA/DTMN/SHDT DR14 14/05/03). Nevertheless, they are included in this document.

2.2.a) Pressure variations and calculations of mean flows

Pressure variations in the 5 containers over time during testing are illustrated in figure 5.

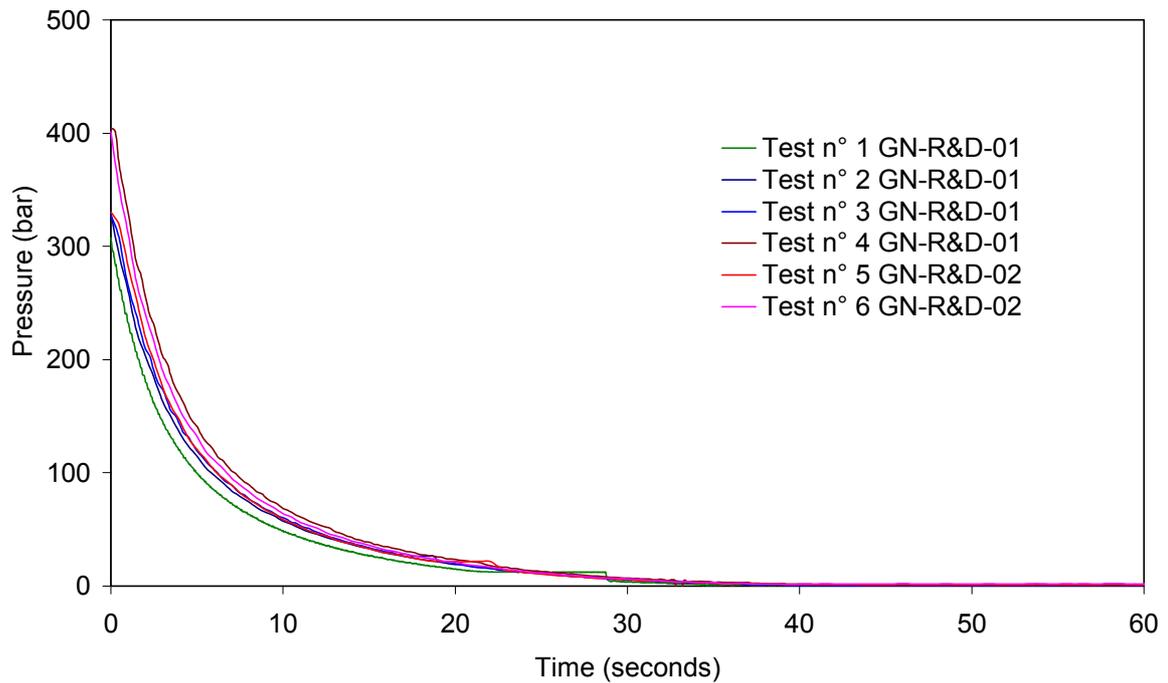


Figure 5. Pressure variations.

In Figure 5, it can be seen that it took between 10 and 12 seconds for pressure to fall from 330 bar to 50 bar and that complete transfer took less than 35 seconds.

Mean flow-rates were calculated, for each test, during transfer at 50 bar and at 10 bar (Table 1).

Table 1. Mean flow-rates for each test

	Pressure (bar)	Temperature (°C)	Time (mn)	Volume H ₂ (TPN L)	Flow-rate (TPN L/mn)	Flow-rate (TPN m ³ /h)
Test n°1 GN-R&D-01	307	16	0	11,988	-	-
	50	16	0.16	2,286	59,400	3,564
	12	16	0.48	567	23,835	1,430
Test n°2 GN-R&D-01	329	16	0	12,677	-	-
	51	16	0.18	2,303	56,585	3,395
	10	16	0.43	458	28,198	1,692
Test n°3 GN-R&D-01	327	25	0	12,269	-	-
	51	25	0.19	2,230	52,377	3,143
	10	25	0.43	462	27,781	1,667
Test n°4 GN-R&D-01	404	28	0	14,447	-	-
	51	28	0.21	2,219	57,543	3,453
	10	28	0.45	450	31,105	1,866
Test n°5 GN-R&D-02	330	24	0	12,404	-	-
	49	24	0.19	2,154	53,478	3,209
	10	24	0.43	443	28,144	1,689
Test n°6 GN-R&D-02	401	23	0	14,559	-	-
	51	23	0.20	2,258	61,505	3,690
	10	23	0.43	460	32,536	1,952

Comparison of tests n° 2, 3 and 5, performed at equivalent initial pressure, reveals that mean calculated flow-rates are similar. The deviations noted may be explained by slight initial pressure differences or by ambient temperature differences.

2.2.1 Connector temperature variations.

During transfer, the temperature of the connector mounted in the protective container placed under rough vacuum dropped by up to 10 °C. Temperature continued to fall for 8 minutes after the test down to negative temperatures, between -5 and -10 °C, downstream from connector and between 0 and 5 °C upstream. The higher temperatures recorded upstream compared to downstream may be explained by the fact that the thermocouple was very close to the confinement tank wall. Line cooling could therefore have been attenuated by diffusion into the wall material. Return to initial temperature took approximately 2 hours.

2.2.2 Calculation of leak rates

Leak rate measurements taken during testing within the confinement tank did not reveal significant gas leaks (**leak rate < 0.024 cm³/mn**). Therefore, the connectors - validated for usage under 200 bar natural gas on vehicles - did not fail, despite being used under 400 bar hydrogen. Leaks detected could not attributed solely to the connector, but also to the various connective parts used in the confinement.

These tests, that revealed satisfactory behaviour of the STÄUBLI connectors tested, were followed by a second test phase. During the second phase, connectors were fitted to a 9-litre tank simulating a vehicle fuel tank.

3 SECOND TEST PHASE

3.1 Test means

The experimental apparatus included some of the first test phase installations. It comprised five 10-litre tanks, a connector and also a 9-litre tank made of a polymer liner (polyamide) and composite material over-wrap (Figure 6).

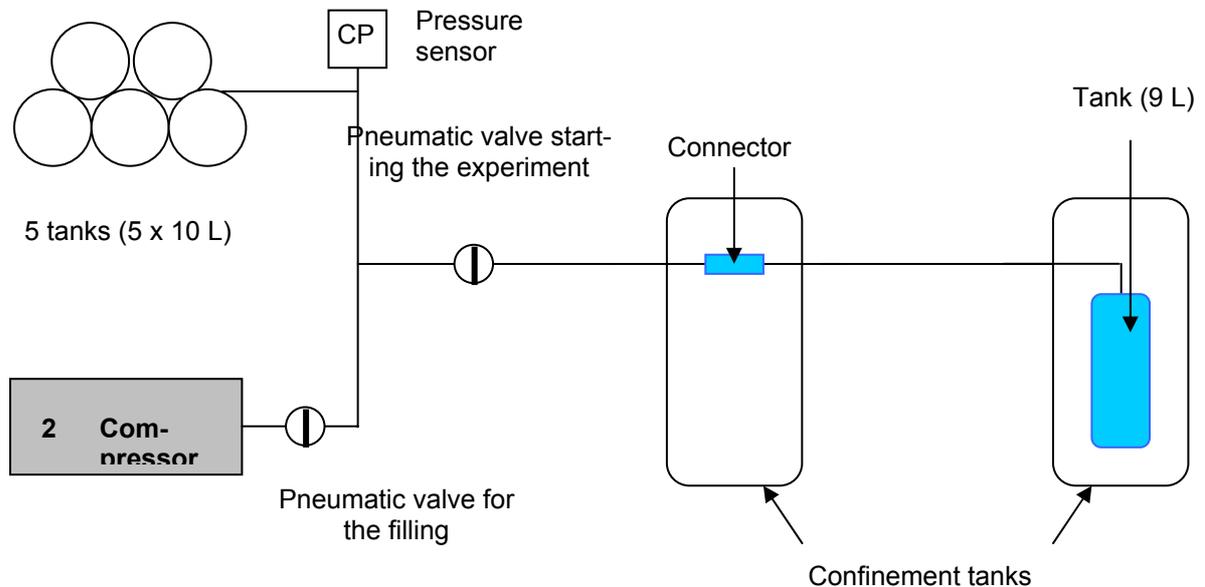


Figure 6. Apparatus schematic diagram.

The five 10-litre containers were first filled under a pressure of 370 bar. The test was then started by opening a pneumatic valve that placed the pressurised containers in communication with a connector and a 9-litre tank installed in confinement tanks. Only the confinement tank containing the connector was placed under vacuum, the 9-litre tank confinement was at atmospheric pressure. During experiments, 9-litre tank and connector temperature and pressure variations were recorded. Temperature was measured on the external surface downstream from the connector as well as at three different heights inside and outside the 9-litre tank (Figure 7). Temperature variations inside the tank were monitored by three thermocouples placed close to the tank neck (TC1), at the centre (TC2) and at the bottom of the liner (TC3). Three other thermocouples were installed outside the tank: on the neck (TC4), at the centre (TC5) and at the bottom of the tank (TC6).

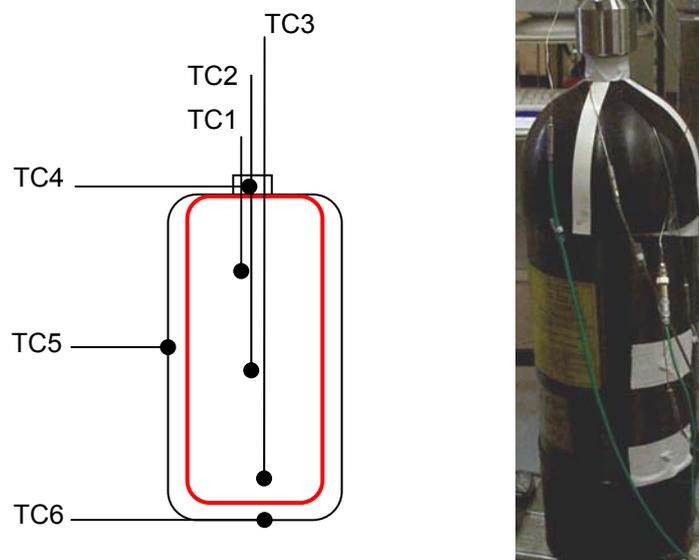


Figure 7. Means used to monitor tank temperature.

During the second phase, four connectors were tested: the GN-R&D-01 and GN-R&D-02 connectors, and also the H2-R&D-01 and H2-R&D-02 connectors (prototypes purposely developed by STÄUBLI).

3.2 Experimental results obtained with GN-R&D-01 and GN-R&D-02 connectors

3.2.1 Pressure variations and mean flow-rate calculations

Pressure variations recorded while filling the 9-litre tank with hydrogen during the various tests are shown in figure 8.

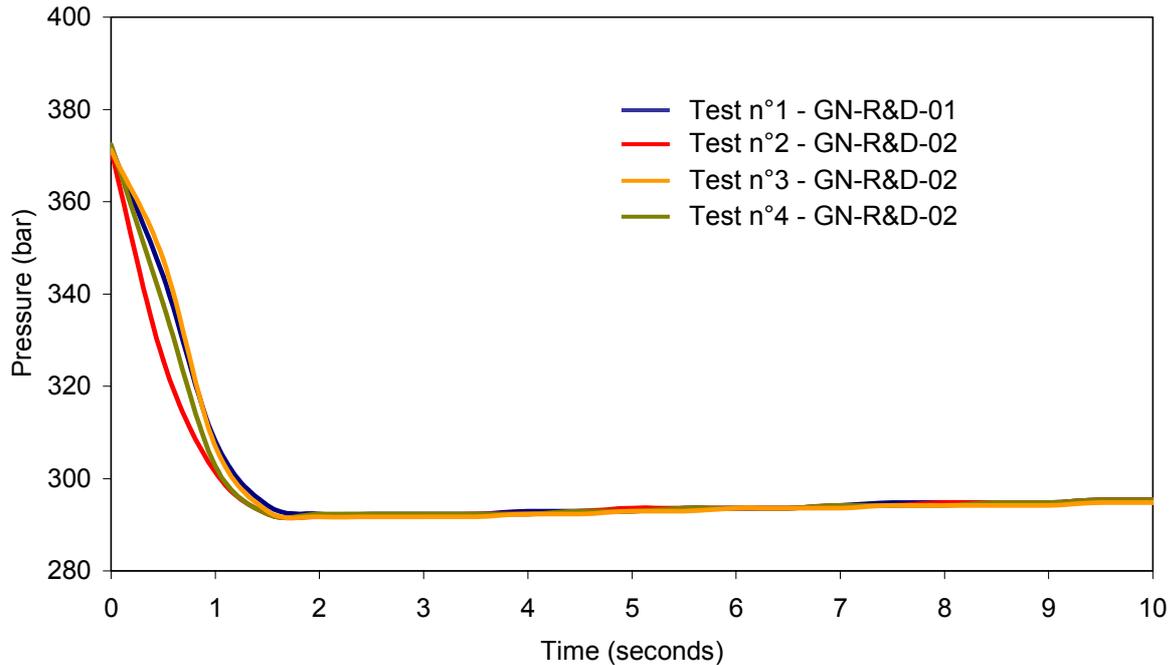


Figure 8. Pressure variations over time.

The transfer took less than 2 seconds. Pressure dropped from 370 bar (initial pressure) to approximately 292 bar. It then increased by 8 bar after one minute due to a temperature increase in the 9-litre tank caused by compression of hydrogen (paragraph 3.2.c). Subsequently, the pressure dropped once more, down to 295 bar at the end of the experiment when the gas temperature inside the 9-litre tank was equivalent to ambient temperature.

The hydrogen filling flow-rate for the 9-litre tank was calculated for each test (Table 2).

Table 2. Mean flow-rate for each test.

	Pressure (bar)	Temperature (°C)	Time (s)	Volume H ₂ (TPN L)	Flow-rate (TPN L/mn)	Flow-rate (TPN m ³ /h)
Test n°1	371	28	0	13,489	-	-
GN-R&D-01	292	28	2	11,077	72,360	4,342
Test n°2	371	26	0	13,560	-	-
GN-R&D-02	292	26	2	11,138	72,660	4,360
Test n°3	371	25	0	13,595	-	-
GN-R&D-02	292	25	2	11,168	72,810	4,369
Test n°4	372	23	0	13,707	-	-
GN-R&D-02	292	23	2	11,238	74,070	4,444

Due to the small number of tests performed and temperature variations between tests, it is difficult to compare flow-rates between two connectors. However, it may be noted that the order of magnitude of the flow-rates calculated for these two connectors are comparable.

3.2.2 Connector temperature variations

Temperature variations downstream from the GN-R&D-02 connector during an experiment are illustrated in Figure 9.

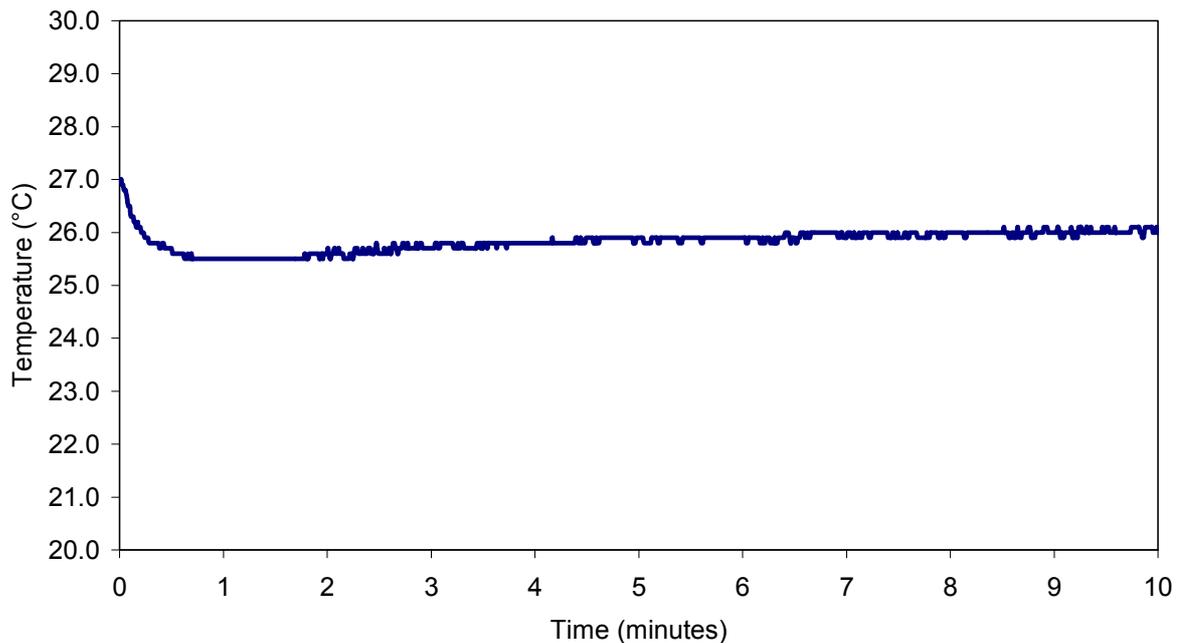


Figure 9. Connector temperature variation.

It was noted that the temperature downstream from the connector fell by 1.5 °C after hydrogen passed through the connector. This drop in temperature could have been caused by circulation of hydrogen at a temperature lower than that of the connector. In fact, it is possible that connector temperature fell following depressurisation of hydrogen from the 5 containers. Furthermore, the 5 containers holding the hydrogen and the connector were not installed in the same room. Consequently, it could also be argued that initially, the temperature of hydrogen in the 5 containers and in the connector during testing were not identical.

3.2.3 Temperature variations inside the 9-litre tank

Temperature variations inside the 9-litre tank over time during a test performed with the GN-R&D-01 connector are shown in Figure 9.

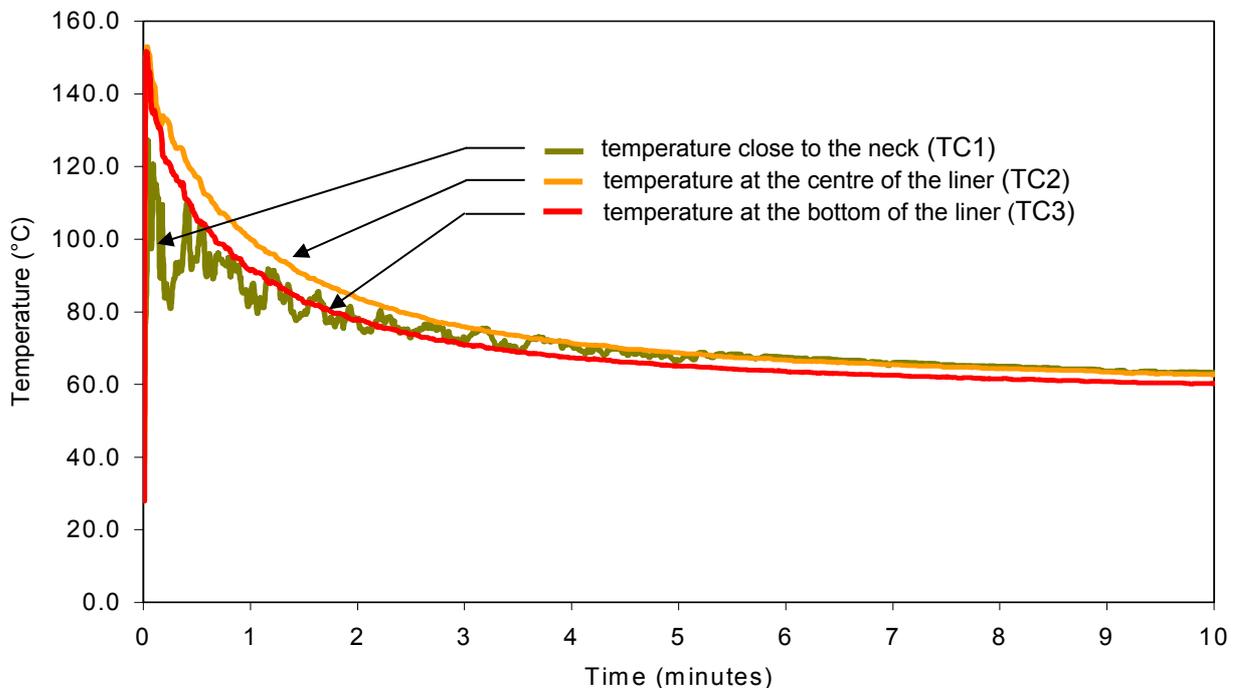


Figure 10. Temperature variations inside the 9-litre tank.

The temperature inside the 9-litre tank increased during testing up to a peak at 150 °C and then fell back to the initial temperature over time. Return to ambient temperature took approximately 3 hours. Tests with the GN-R&D-02 connector yielded identical results.

In Figure 10, it may be noted that the temperature recorded near the tank neck oscillates appreciably during first minutes of the test. This temperature variation is definitely attributable to the position of the TC1 thermocouple that is directly subject to hydrogen flow during filling.

Furthermore, it may be noted that the temperature measured at the centre by thermocouple TC2 was slightly higher than that measured at the bottom of the liner by thermocouple TC3. This difference may be explained by the fact that thermocouple TC3 measuring the temperature at the bottom of the liner was, on the contrary to TC2, in contact with the liner.

3.2.4 Temperature variations outside the 9-litre tank

Temperature variations recorded outside the 9-litre tank over time during a test performed with GN-R&D-02 connector are illustrated in Figure 11.

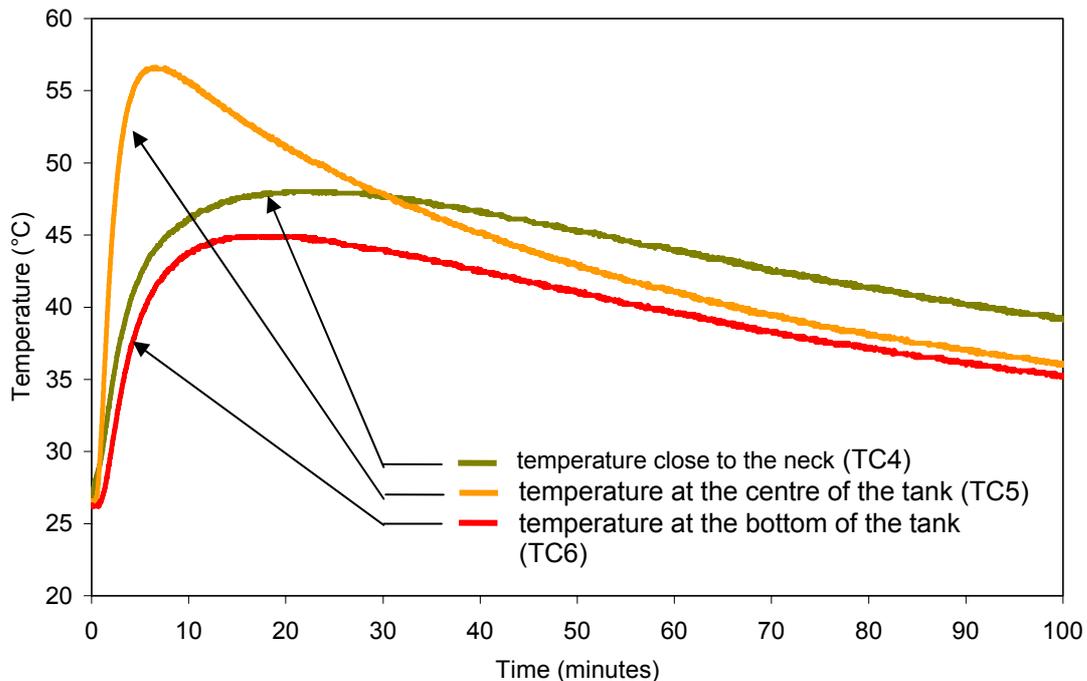


Figure 11. Temperature variations outside the 9-litre tank.

Temperature variations outside the tank are not identical at every point. Neck temperature increased by 3 °C during the first minute of the experiment, probably due to heating caused by hydrogen passing through the neck at a high flow-rate. After this time period, neck temperature continued to rise, but more significantly due to the heat present in the tank, up to a maximum temperature of 48 °C after about 20 minutes. Temperature at the centre of the tank remained stable for 30 seconds and then rapidly increased for 6 minutes due to the transmission of heat generated by compression of hydrogen through the tank via conduction. The maximum temperature recorded at the centre of the tank

was 57 °C. The temperature at the bottom of the tank remained constant for 1 minute, and then increased to a maximum temperature of 45 °C after 20 minutes. Temperature variations noted outside the tank during tests with the GN-R&D-01 connector were comparable.

Differences between temperatures measured at the tank surface may have been due to the fact that the tank is made of several materials. It comprises a polyamide liner over-wrapped with filaments of glass- and carbon- fibre based composite materials and an aluminium neck (AA 6061 T6). Therefore, the neck would not conduct heat in the same way as the liner that is fully over-wrapped with composite materials. Furthermore, the thickness between the internal and external wall of the tank is not the same at every point, this could induce behavioural differences even if the material was identical. Lastly, convection could have taken place within the confinement tank and this could have had an effect on temperature variations at the surface of the tank.

Following these experiments, proof-testing was performed on the H2-R&D-01 and H2-R&D-02 connectors. For this analysis, the 9-litre tank along with the thermocouples mounted on the tank and the connector were replaced. Furthermore, the temperature upstream from the connector was monitored during testing.

3.3 Experimental results obtained with the H2-R&D-01 and H2-R&D-02 connectors

3.3.1 Pressure variations and calculation of mean flow-rates

Pressure variations during experiments are illustrated in Figure 12.

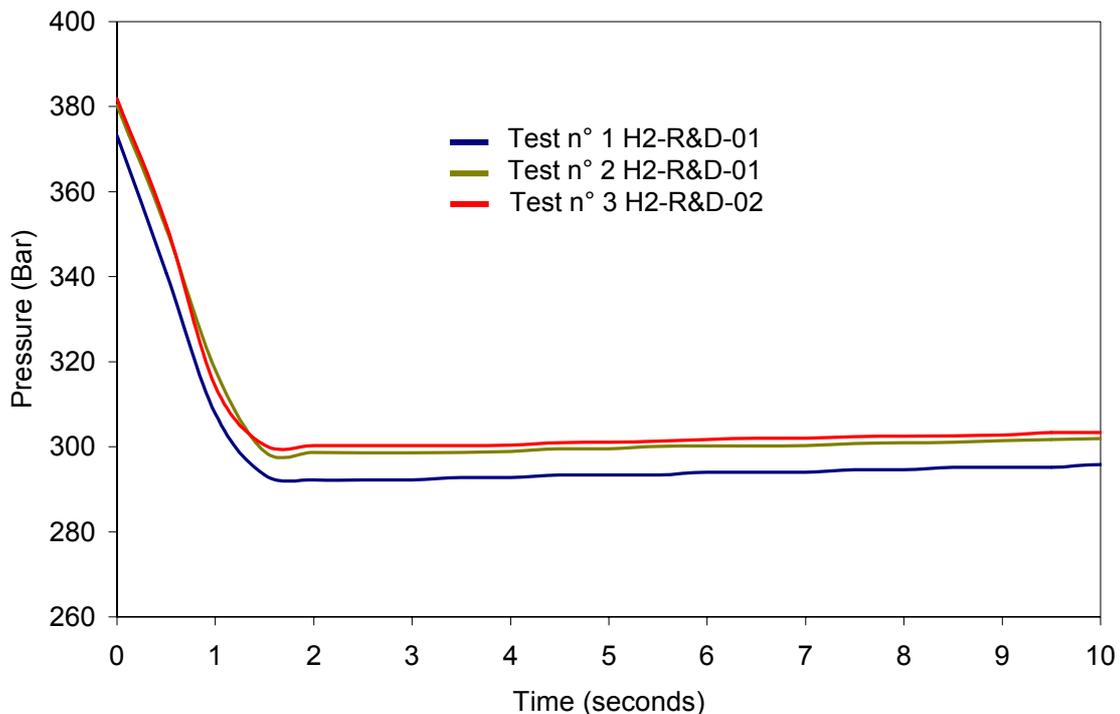


Figure 12. Pressure variations over time.

Pressure variations were comparable to those observed with the GN-R&D-01 and GN-R&D-02 connectors (paragraph 3.2.a).

Transfer took less than 2 seconds. Pressure dropped while filling the 9-litre tank and then increased by 8 bar after one minute. Subsequently, pressure fell again to 292 bar at the end of the experiment.

Mean hydrogen flow-rate during filling of the 9-litre tank was calculated for each test (Table 3).

Table 3. Mean flow-rate for each test.

	Pressure (bar)	Temperature (°C)	Time (s)	Volume H ₂ (TPN L)	Flow-rate (TPN L/mn)	Flow-rate (TPN m ³ /h)
Test n°1	373	17	0	13,984	-	-
H2-R&D-01	292	17	2	11,450	76,020	4,561
Test n°2	380	17	0	14,192	-	-
H2-R&D-01	299	17	2	11,679	75,390	4,523
Test n°3	382	18	0	14,251	-	-
H2-R&D-02	300	18	2	11,711	76,200	4,572

The order of magnitude of the mean calculated flow-rates for these tests was comparable to that obtained for GN-R&D-01 and GN-R&D-02 connectors (Table 2).

3.3.2 Connector temperature variations

Temperature variations downstream and upstream from the H2-R&D-01 connector over time are illustrated in Figure 13.

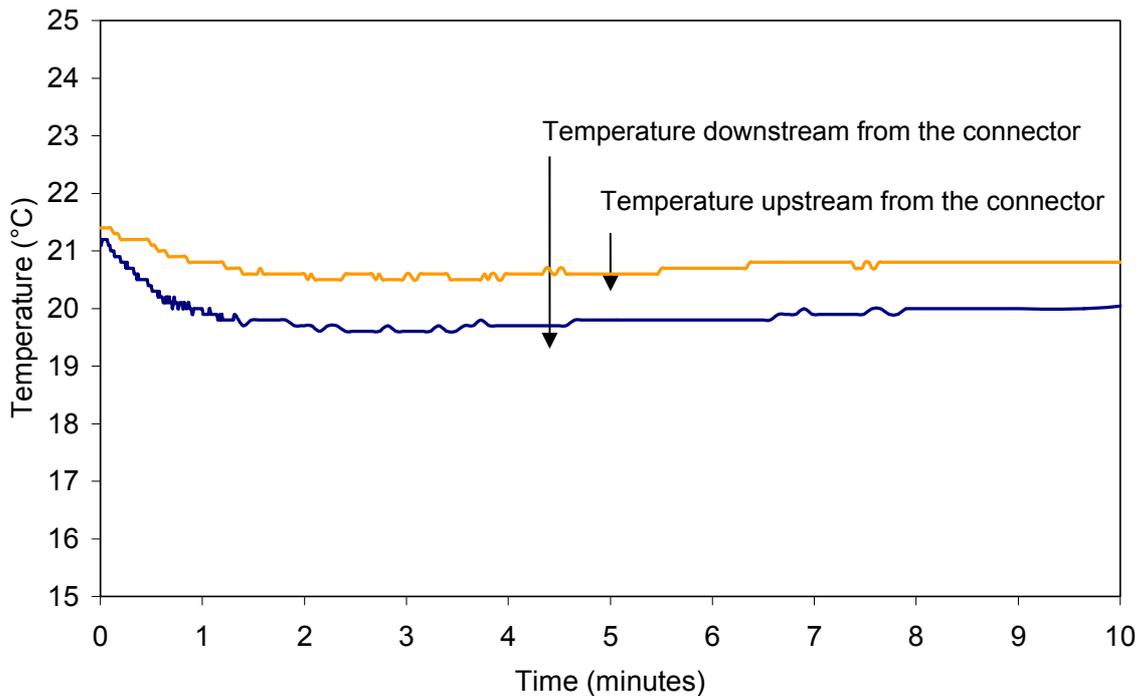


Figure 13. Temperature variations upstream and downstream from the connector.

Once again, the temperature drop at the connector was more significant downstream than upstream due to the proximity of the confinement tank metallic wall. Irrespective of connector, the upstream temperature fell by 1 °C after hydrogen passed through the connector while the downstream temperature fell by a maximum of 1.5 °C.

3.3.3 Temperature variations inside the 9-litre tank

Temperature variations inside the 9-litre tank during a test performed with connector H2-R&D-01 are provided in Figure 14.

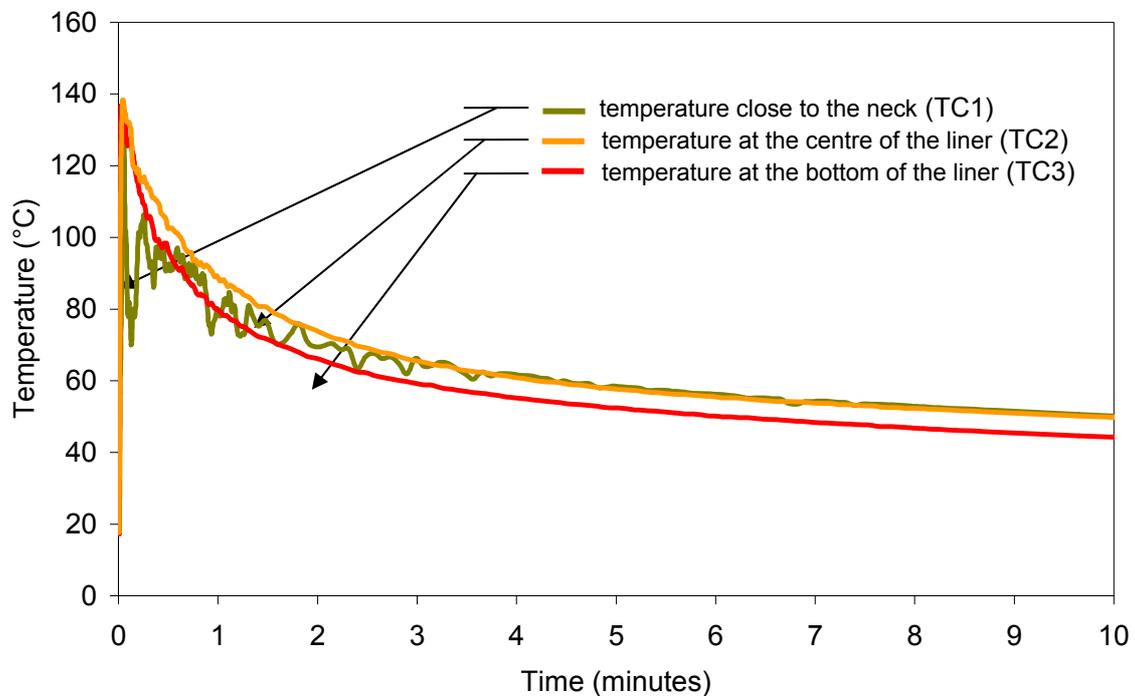


Figure 14. Temperature variations inside the 9-litre tank.

The temperature inside the 9-litre tank increased up to a maximum value of 140°C and then, over time, fell back to the initial temperature. Return to ambient temperature took approximately 2 hours. The temperature inside the tank varied in a manner comparable to the that noted during tests with connector H2-R&D-02.

It may be observed that the temperature measured near the tank neck always oscillates more significantly during the first minutes of the test and that the temperature measured at the centre is higher than that measured at the bottom of the liner.

3.3.4 Temperature variations outside the 9-litre tank

Temperature variations outside the 9-litre tank measured over time during the test performed with H2-R&D-02 are shown in Figure 15.

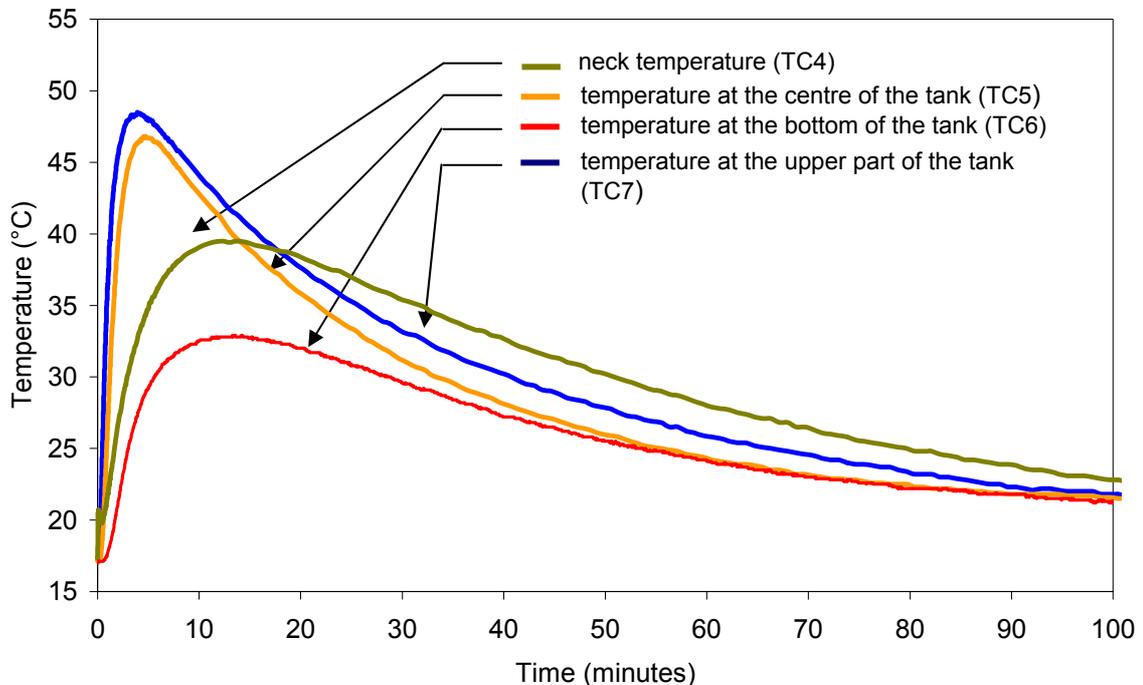


Figure 15. Temperature variations outside the 9-litre tank.

Temperature variations outside the tank noted during tests with connectors H2-R&D-01 and H2-R&D-02 are quite similar to those observed with GN-R&D-01 and GN-R&D-02. Deviations that may be noted are probably caused by replacement of thermocouples and the fact that experimental conditions were not exactly the same. Neck temperature increased by 3 °C while the 9-litre tank was being filled and then fell by less than 1 °C during the 30 first seconds of the experiment. It then increased again up to a maximum temperature of 40 °C after 12 minutes. Temperature at the centre of the tank remained stable for 20 seconds and then increased rapidly over 5 minutes to a maximum value of 47°C. Temperature at bottom of the tank remained constant for 1 minute and then increased to a maximum temperature of 33 °C after 12 minutes.

In addition to thermocouples used in experiments performed with the GN-R&D-01 and GN-R&D-02 connectors, another thermocouple (TC 7) was mounted on the upper part of the tank (Figure 16).

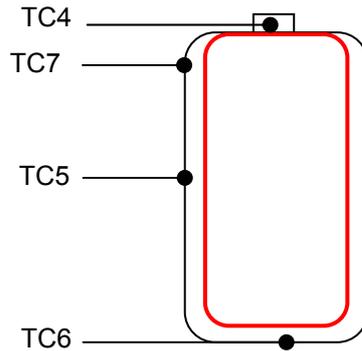


Figure 16. Position of thermocouples used to monitor external tank wall temperature.

Temperature at this point of the tank varied in a manner comparable to that observed at the centre of the tank. The temperature remained stable for 10 seconds and then increased over 3.5 minutes to reach a maximum temperature of 48 °C.

4 CONCLUSION

The main aim of this study was to analyse the behaviour of the various STÄUBLI connectors when subject to high pressure hydrogen. Firstly, two connectors designed and marketed to transfer natural gas to vehicles under a pressure of up to 200 bar, were tested with venting. These tests notably revealed the effect of hydrogen depressurisation on connector temperature and illustrated that these connectors did not fail despite use under 400 bar hydrogen. Following these experiments, a second test series was performed on these connectors and two other connectors purposely developed by STÄUBLI to fill tanks with hydrogen. During this second test phase, four connectors were interfaced with a 9-litre tank simulating the vehicle fuel tank. The results obtained for each connector were comparable. The 9-litre tank was filled to a pressure of 300 bar in less than 2 seconds. Following transfer of hydrogen, connector temperature dropped slightly (by approximately 1 °C). We also noted that filling the tank in a few seconds caused the temperature within the tank to rise significantly. This increase in temperature entailed a more moderate increase in temperature outside the tank and that, in the framework of this study, was not identical at every point of the tank. The significant increase in temperature within the tank could lead to deterioration of the polymer liner. Therefore, the filling flow-rate needs to be controlled in order to avoid causing damage to the liner that could otherwise impugn tank leak-tightness.