External Ortho-para Converter of the NPDGamma LH2 Target

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**External Ortho-para Converter (OPC)**

The NPDGamma experiment needs the liquid hydrogen in the target to be in the “para” molecular state. Hydrogen molecules exist in two distinguishable states: “ortho”, with the spins of the proton parallel, and “para”, with the spins of the protons antiparallel, and at room temperature the gas is 75% ortho and 25% para. To catalyze the conversion between these two molecular states requires a magnetic material with large surface area. The energy difference between these two states is about the same as the heat of vaporization per molecule of hydrogen. The experiment therefore needs an external OPC to shorten the filling time for the liquid parahydrogen target to a manageable time.

An external O/P converter will be constructed in which the incoming H2 gas flows through a tube with catalyst cooled by a mechanical refrigerator. The idea is to use this external OPC to perform much of the ortho-para conversion of the H2 gas before it is liquefied in the target cryostat and thereby reduce the filling time of the system by about a factor of 2.
Figure 1: Left: 3D rendering of proposed external OPC showing the mechanical refrigerator, a flange with welded Conflat penetrations to conduct the gas into the OPC tubing inside the vacuum chamber, and the tubing holding the converter powder coiled around the refrigerator. Right: Picture of the vacuum chamber for the refrigerator.
Figure 2: Drawing of the flange for the mechanical refrigerator and the hydrogen gas inlet and outlet. Conflat flanges will be welded to the flange.
Figure 3: mechanical refrigerator for the external OPC. Copper foil is used to connect the two stages of the refrigerator to ensure that the temperature of the coldest stage of the refrigerator stays well above the 20K required to liquefy hydrogen at the gas supply pressure.
Figure 4: Section of gas handling system and relief system located inside the ventilated cabinet which includes the external OPC. When operating the OPC, the valve MV124 is closed and the hydrogen gas from MV125 supply line flows into and out of the OPC and then into the FR100 flow restrictor and toward the hydrogen fill/vent line.
General Description of External OPC Design and Operation

The supply line from the hydrogen supply manifold is connected to a gas handling system (GHS) located close to the BL13 enclosure. The gas goes through a liquid nitrogen trap (CT) in the GHS panel to remove water and reduce other contaminants. Then the cleaned H2 gas enters an ortho-para conversion chamber (OPC) based on ferric oxide powder and operated at 30K for partial conversion of the gas before entering the refrigerator. We plan to use hydrous ferric Fe(OH)$_3$ converter with the mesh size of 30x50. The hydrogen passes through a coiled tube which contains the converter material. The stainless steel tubing is soldered onto a copper annulus for thermal contact with the refrigerator. The thermal connection between the copper cylinder and the refrigerator is designed in such a way that the refrigerator can be disconnected from the OPC volume without the need to disconnect the OPC from the top flange of the vacuum chamber. The Fe(HO)$_3$ is prevented from leaving the annular region with fine wire mesh on the inlet and outlet tubes. The OPC is cooled by a mechanical refrigerator (an Austin Scientific cryocooler (model # M1020-CS) along with a CTI Cryogenics 8500 compressor). Temperature of the refrigerator will be monitored using diode thermometry. A heater will surround the tubing housing the catalyst to regenerate the catalyst. The maximum gas flow rate, liquefying rate, which is determined by the cooling power of the refrigerators, and the properties of hydrogen (see below), will be 10-20 standard liters/minute.

Path of the Hydrogen gas through the OPC

The hydrogen gas comes from the MV125 hydrogen supply line. It then encounters the closed valve MV124 and flows in a SS line toward the OPC. The gas then sees the following items: (a) the first 1.33” Conflat flange seal connecting the incoming hydrogen line to a penetration in a flange located on top of a vacuum chamber that houses the OPC and its associated cryostat, (b) a weld penetration in the flange, (c) a second 1.33” Conflat flange seal connecting the hydrogen fill line to the internal coiled section of tubing used as the container for the OPC catalyst material, (d) an internal mesh to contain the OPC powder, (e) the coiled SS tubing of the OPC, which wraps around a copper cylinder which is in good thermal contact with a mechanical refrigerator used to lower the temperature of the OPC material, (f) a second internal mesh near the exit of the line, (g) a third 1.33” Conflat flange seal connecting to the outgoing hydrogen to a second penetration in the flange, (h) a fourth 1.33” Conflat flange which connects the outlet of the OPC to the downstream portion of the hydrogen fill line. The gas then encounters a flow restrictor FR100 on its way to the hydrogen target fill/vent line.
Safety Features of the Design/Mitigation of Potential Hazards

The introduction of an external OPC into the hydrogen line does not change the safety of the target system as long as the OPC is not the weakest point in the line. Hazards have been identified and mitigated as follows:

(1) **Leak in the hydrogen line entering and exiting the OPC.** Hazard: hydrogen gas leaks from the tubing and demountable joints leading into and out of the OPC. **Mitigation:** The hydrogen gas will be conducted through the OPC system using SS tubing with welded components. Conflat feedthroughs welded to the external flange of the vacuum chamber of the OPC conduct the hydrogen through the vacuum flange, into the internal SS tubing which contains the converter material, and out again to the hydrogen line that feeds the fill/vent line of the target. Conflat flanges of 1.33” diameter are chosen based on the results of out test data on their behavior at temperatures below room temperature and under internal pressure. The possibility of a leak is reduced by the use of welded joints, the use of CF flanges for all demountable portions, and proper procedures for installation of the CF joints. Helium leak testing will be required in the operating procedure to be performed before introduction of hydrogen gas into the OPC system. If there is a leak outside the OPC, it is limited to less than 25 slm from other portions of the gas handling system and is handled by the cabinet ventilation and detected by H2 sensors in the cabinet.

(2) **Prevention/mitigation of the possibility of liquification of hydrogen in the OPC.** Hazard: the hydrogen gas flowing into the OPC is liquefied inside the converter volume, thereby creating liquid hydrogen in an unintended location. **Mitigation:** The unmodified cryocooler has a base temperature that is low enough to liquefy the hydrogen. We propose to prevent this by introducing a passive, mechanical thermal short between the first and second cold stages which keeps the temperature of the coldest stage above 30K. Test measurements (see below) have established that this temperature, which is several degrees above the temperatures at which hydrogen can liquefy at gas pressures present in the hydrogen fill line, is stable over long periods of operation. It is possible that over long periods of time the thermal contact between the two cold stages which defines their base temperatures might slowly change (through, say, slow relaxation of the force exerted by the bolts on the thermal link). The bolts will be secured by lock washers to reduce the probability of such relaxation, and thermal conducting grease (Apiezon) will be used in the area of thermal contact to help make the area of thermal contact defining the base temperarture of the cold stages more insensitive to small changes in the force of the bolts. Diode thermometers on the coldest portion of the OPC will be used to monitor the temperature to ensure that any such possible change in the thermal contact between the stages can be observed.

(3) **Rise in gas pressure in the OPC** Hazard: the pressure of the cold hydrogen gas in the OPC volume undergoes a rapid increase, thereby causing pressure changes in other areas of the gas handling system and confusing operators, leading to possible operator error. **Mitigation:** Such a pressure rise could be caused in
various ways: for example (a) a failure of the refrigerator during operation, (b) a plug in the fill line, (c) a leak in the main vacuum chamber, thereby changing the temperature and therefore pressure of the cold gas in the OPC, (d) an accidental warming of the cold gas in the OPC due to operator error with the heater needed to regenerate the OPC converter material, (e) failure of the pump which evacuates the vacuum chamber. The inlet and outlet lines for the hydrogen lines will possess relief valves (RV105 and RV106 in the drawing) which are connected to the main vent system. The heater which is present to regenerate the catalyst will be physically disconnected during hydrogen fills to reduce the possibility of operator error causing a transient pressure increase through an accidental warming of the cold gas in the converter area. The helium leak check and the removal of the heater connection before introduction of hydrogen gas into the OPC will be included in the operating procedure. The potential for a plug in the fill line is reduced by the upstream cold trap CT. The pump that evacuates the vacuum chamber will be vented to the hydrogen vent stack and during operation the pump will be valved off from the chamber.

(4) **Rupture/leak of the fill line in the OPC.** Hazard: release of hydrogen gas into the vacuum chamber of the OPC through rupture of the tubing or the demountable joints. One could imagine that various possibilities mentioned in (3) could lead to an internal pressure in the hydrogen line that is high enough to breach the fill line or to induce a leak in the Conflat flange connections. **Mitigation:** In addition to the features of the design mentioned in (3), others reduce further the safety consequences of (4). A hydrogen sensor (International Sensor Technology) is placed inside the cryostat vacuum chamber to detect any hydrogen leak which would presumably be the first sign of such an event. Pressure gauges on the main vacuum chamber are present which would indicate any such large leak occurring inside the vacuum chamber. If such a leak does occur, the vacuum chamber is protected from excessive pressure by a 2 psid check valve CV105. Conflat flanges of the same size as those on the vacuum chamber and ceramic electrical feedthroughs have been previously leak tested with helium gas using greater internal pressures without a leak. Finally, should the leaking hydrogen gas escape the vacuum chamber, the entire OPC including the vacuum chamber is located inside a ventilated cabinet.

(5) **Blockage of the fill line.** Hazard: a blocked fill line upsets the steady-state nature of normal hydrogen target filling and thereby requires operator action, leading to an increased possibility of error. **Mitigation:** Blockage of the fill line is made unlikely by the liquid nitrogen cold trap upstream of the OPC. Should the OPC become blocked despite this precaution, the relief valves RV105 and RV106 will protect against overpressures. The operating procedure will include a clear set of actions to be taken in the event of a blockage in the line involving the action of pumps in the gas handling system, the valve MV124, and the heater. The effectiveness of the proposed operating procedure will be tested with neon gas to confirm that the procedure is safe and effective.
Relief valve settings and functions

RV105

**Function:** relieve an over-pressure condition in the external OPC chamber.
**Location:** between MV125 and MV124 at the entrance to the external OPC.
**Set point:** 100 psid cracking pressure
**Reasoning:** If MV126 must be throttled down during filling, this setting is sufficiently high to avoid venting H2 unnecessarily and adequately below the yield strength of the external OPC chamber.

RV106

**Function:** relieve an over-pressure condition in the H2 supply system downstream of the external OPC.
**Location:** between MV124 and MV126 at the exit of the external OPC
**Set point:** 100 psid cracking pressure
**Reasoning:** This set point avoids the possibility of over-pressuring the H2 supply line.

External OPC Instrumentation

Table 1. Instrumentation associated with external OPC.

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Transducer requirements</th>
<th>Operating range</th>
<th>Accuracy</th>
<th>Mechanical data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermometers</td>
<td>o-p converter, cryorefrigerator</td>
<td>10-300 K on converter, first and second stage of refrigerators,</td>
<td>0.2 K accuracy in 10-30 K range, 2 K accuracy in 70-300 K range</td>
<td>0.1 K in 10-30 K range, 2 K in 70-300 K range</td>
</tr>
<tr>
<td>Pressure gauges</td>
<td>External OPC vacuum chamber, hydrogen fill line</td>
<td>2-&gt;10^{-7} bar on main vacuum and LH2 target, 2-&gt;10^{-3} bar on He jacket</td>
<td>3% near atmospheric pressure</td>
<td></td>
</tr>
<tr>
<td>Heaters</td>
<td>o-p converter</td>
<td>0 to 25 W</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hydrogen sensors</td>
<td>Vacuum chamber</td>
<td>0-100% LEL</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Test Results

5.4 Low-temperature CF Seals

There are four 1.33” diameter stainless steel Conflat (CF) flanged seals with copper gaskets in the design of the external OPC as shown in Figure 1. The external pair of demountable joints are included for assembly/disassembly of the OPC unit as a whole, and the internal pair of joints are included to provide a means to fill and extract the OPC
converter material. These Conflat flanges may reach temperatures lower than room temperature. The CF seals are used in other operating LH\textsubscript{2} targets (at Thomas Jefferson Lab, for example) and are known from their experience to be reliable at cryogenic temperatures if properly used. We have done an exhaustive investigation of their reliability at low-temperatures and under internal pressures. The test results are available on web side www.iucf.indiana.edu/U/lh2target/export-files/ and are summarized in the next section.

5.4.1 Testing of Conflat and VCR Seals by Thermal Cycling and under Pressure

Problem: Confirm the reliability of the CF flanged joints at low temperatures (LN\textsubscript{2} temperatures) and under internal pressure (up to 100-200 psig). Confirm the reliability of the VCR joint at low temperatures and under internal pressure.

Method:
- Carefully make a joint by using a torque wrench and torques defined by the manufacturer
- Carefully leak check the joint when thermal cycled or when the joint still cold.
- Pressurize with helium gas the joint to 100-200 psig inside and leak test the joint.

The detailed description of the procedures and results are available in web page www.iucf.indiana.edu/U/lh2target/export-files/.

As a summary we can say that in the level of $10^{-10}$ Torr liter/s no leaks were found. Using a sniffer with internal pressure no leaks to the outside were seen in the sensitivity of $10^{-7}$ Torr liter/s.

10.4.1 Test of the minimum temperature of the external OPC refrigerator

Date: November, 2007
DUT: external OPC refrigerator
Object: verify that thermal connection between refrigerator stages can keep temperature of second stage above 21K.

We modified a cryogenic refrigerator to be used with the external ortho-to-para converter (OPC) for the liquid hydrogen target of the NPDGamma experiment. An Austin Scientific cryocooler (model # M1020-CS) was used, along with a CTI Cryogenics 8500 compressor. The cooling section of the cryocooler was sealed in an airtight aluminum box, evacuated by roughing and turbo pumps. Two Lake Shore four-lead silicon diode thermometers were used to measure the temperature. The thermometers were calibrated at room temperature and at liquid nitrogen temperature and the standard Lakeshore calibration curve for silicon diodes was used to infer the temperatures.

We first ran the cryocooler unmodified and measured the temperature at both stages which read 53 K and 17 K. In order to raise the temperature of the second stage we installed a thermal coupling between the stages. One continuous piece of 0.14 inch thick copper foil was pressed flat against the surface of the two heat stations to connect them thermally. In order to occupy any gaps between the surface of the cryocooler and the
copper foil, Apiezon H grease was used. After cooling down again, the temperatures of
the first and second stages read 46 K and 30 K. The refrigerator was operated
continuously for a week and the temperatures remained stable. The temperature is
therefore sufficient to perform ortho-para conversion but is still warm enough to prevent
any of the hydrogen from liquefying.

Test Performed By:                        Approved By:
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