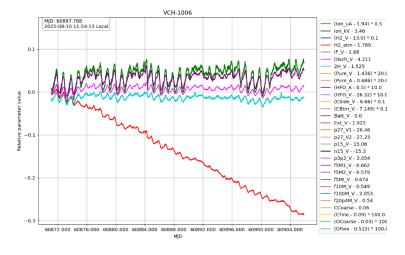


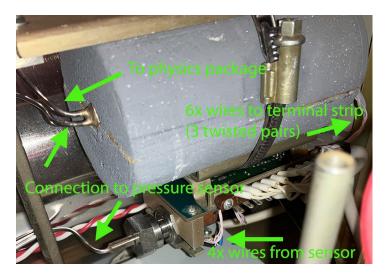
Prior experience had shown that cranking up the temperature in the canister containing the lanthanum nickel hydride storage media was a temporary fix at best, so it was time for more drastic measures.

This document recounts an experimental procedure undertaken to replenish the molecular hydrogen reservoir in my VCH-1006 passive maser.

The maser has been working well for over a year, but given that it's almost 25 years old judging by component date codes in the 2000-2001 timeframe, it was clear that it would need a hydrogen refill in the not-too-distant future. In mid-August 2025, the H2 pressure fell below its rated 1.5-atm minimum.







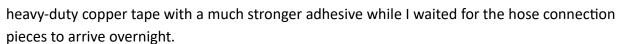
Numerous sources mention that exposing the LaNiHx crystals to atmospheric conditions causes a passivation process to take place that is difficult to reverse, requiring a high-temperature bake under vacuum. Air probably isn't great for the nickel purifier element, either. All of that being said, there was never any possibility of recharging the storage media without opening up the high-pressure H2 plumbing circuit at least momentarily. So the plan was to open it at the only available point – the pressure sensor – and then conclude the business quickly, keeping the line sealed as well as possible while getting the necessary hardware together.

The first step was simply to identify the fitting size and thread pitch so that a compatible filling adapter could be ordered.



After unscrewing the line at the pressure sensor, I immediately covered its end-face aperture with a piece of Kapton tape. I then proceeded to disassemble the bracket and clamp holding the canister in place so that I could measure the threads on the pressure sensor back at the workbench. So far, so good... but at some point the tape came off.

It's possible that the H2 lines were open to air for one to two hours before I noticed the missing tape. I switched to a piece of



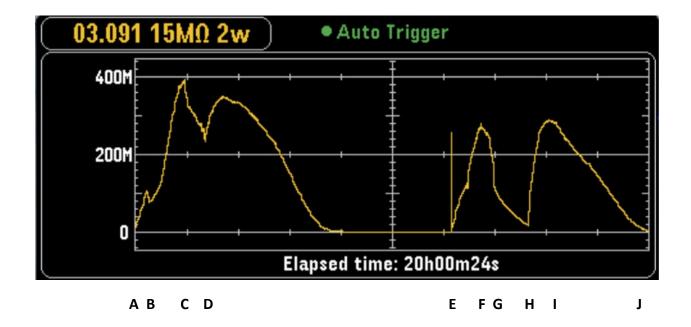


Upon turning on the gas for a trial run at room temperature, my initial impression was unsettling. Most literature indicates that the gas-absorption process is noticeably exothermic, requiring cooling during the refilling process to keep the reaction going. But the canister didn't seem to warm up . A DMM connected to the thermistor at pins 3 and 4 of the associated connector did not indicate any internal temperature rise at all.



The next phase of the plan was to chill the hydride canister with dry ice....

... and after 20 hours of further work, outlined on the next page, the dry ice yielded *much* better results.



A: Start of recorded NTC thermistor readings at pins 3-4 of hydride oven connector, a few minutes after surrounding the canister with several pieces of dry ice. (Higher temperature=lower resistance, normally about 350k at 25C). Initial H2 pressure 240 psi with regulator valve closed.

**B**: Valve opened to enable H2 gas flow. Slight temperature increase noted; added more dry ice to cover the canister completely. External thermocouple reads -20C at this point.

**C**: External thermocouple positioned beneath canister reaches lowest observed reading of -55C. Internal temperature reported by thermistor stops decreasing and begins rising at an inconsistent rate. Bottle pressure reads 180 psi.

**D**: Added substantially more dry ice for overnight recording. Bottle pressure reads 160 psi. Thermistor temperature decreases at first, then begins slow increase lasting several hours.

E: Bottle pressure is now 130 psi. Dry ice ~95% gone; added more. Thermistor resistance shows brief spike artifact, then the temperature starts decreasing again.

F: Another warming cycle begins, similar to C.

G: Fast part of warming cycle ends and temperature begins dropping at rate similar to overnight measurement

H: Dry ice still covers canister, but most gone. External thermocouple reads -35C. Pressure is 125 psi, indicating that about half of the hydrogen (~8.5L) has either been absorbed by the LaNiHx powder or has leaked somewhere. Leakage seems less likely given the inconsistent rate of pressure decrease over time, though. Added more dry ice.

I: Thermocouple reading falls to -50C over the next few minutes. Bottle pressure unchanged at 125 psi. Temperature bottoms out and starts to increase once more.

J: Four hours later, about half of the dry ice is gone. Temperature readings have risen slowly over this period, and bottle pressure has remained stable at 125 psi for several hours.

The maser's performance characteristics post-refill were unchanged in all respects. Its output frequency was high by about +2E-14, just as it had been earlier. This is a good sign, as the sensitive dependence of frequency on hydrogen flux suggests that the nickel purifier is none the worse for wear. Another good sign is that the ion pump current has remained where it was at about 2 microamps.

It does seem likely that the hydrogen storage media has undergone some loss of activity due to exposure to atmospheric oxygen and moisture, though. I don't know whether to expect the refill to last a week, a month, or ten years, but the pressure has remained constant over the first 24 hours at least.

If it's necessary to revisit the issue in the future, I'll probably replace the hydride canister with an external H2 bottle. Arguably that would've been the best approach to begin with, in addition to installing a manifold to allow the lines to be purged.

## Components

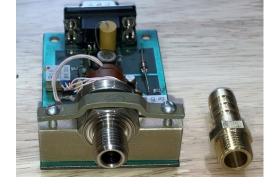
Tank connection hardware (<u>M12-1.25 10mm adapter</u>, <u>3/8"</u> to <u>3/16 reducer</u>, <u>3/16"</u> and <u>3/8"</u> synthetic rubber hoses):



FOCMKEAS 2PCS Brass Hose Barb Fittings, Metric M12-1.25 Male to Barbed Fit hose ID 10mm, Barbed Hose Fitting, Male Thread Hoses Barb Adapter Connector, Hoses



Metaland Brass Hose Barb Reducer, 3/8" to 3/16" Hose ID, Reducing Barbed Hose Splicer Mender Coupler Water Fuel Air (Pack of 2)





3/16 Inch (5mm) ID Fuel Line Hose 10FT NBR Rubber Push Lock Hose High Pressure 300PSI for Automotive Fuel Systems Engines



3/8 Inch (10mm) ID Fuel Line Hose 10FT NBR Rubber Push Lock Hose High Pressure 300PSI for Automotive Fuel Systems Engines

## Hydrogen gas and regulator:

Hydrogen Pure Gas 99.999% in 17 Liter Cylinder CGA 600 Connection



Price: \$54.00

SKU: 17L-83P

CGA: 600

Brand: GASCO

Availability: 7 to 10 Days Production Plus Shipping Time

Dial a Flow Regulator, GASCO Part Number 74-DAF/CGA600, CGA-600 Connection 17L and 34LS Steel Cylinders ONLY



 Price:
 \$299.00

 SKU:
 74-DAF/CGA600

 Valve Inlet:
 CGA 600

 Outlet:
 3/10\* Hose Barb

 Flow Rate:
 0.3, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 5.0, 6.0, 7.0, 8.0

 Gauge PSI:
 600 PSI

 Material:
 BRASS, NICKEL PLATED

 Shipping:
 Froe

 Brand:
 GASCO

 Availability:
 Ships From Factory lead time 3-5

 Does From Factory lead time 3-5
 Does From Factory lead time 3-5

## **End notes**

- Efforts to contain the dry ice to the immediate vicinity of the hydride canister were not very successful, so it was necessary to remove condensation from various PC boards off before powering the maser back on. These included the oven control board and the supervisor/controller interface assembly beneath the plastic label to the right of it.
- Although nice to have, the "Dial-a-Flow" gas regulator is expensive and not really needed. A simple on-off valve is sufficient when the bottle's fill pressure is only 240 psi, as was the case here.
- When I removed the gas fitting from the pressure sensor, I found that it was almost possible to unscrew it by hand. It's unclear what the proper fastening torque should be, particularly given the tendency of PTFE to cold-flow. I used about 15 lb-in (1.7 Nm) when reconnecting.