

# Full Scale Alternative Catalyst Testing for Bosch Reactor Optimization

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Current air revitalization technology onboard the International Space Station (ISS) cannot provide complete closure of the oxygen and hydrogen loops. This makes re-supply necessary, which is possible for missions in low Earth orbit (LEO) like the ISS, but unviable for long term space missions outside LEO. In comparison, Bosch technology reduces carbon dioxide with hydrogen, traditionally over a steel wool catalyst, to create water and solid carbon. The Bosch product water can then be fed to the oxygen generation assembly to produce oxygen for crew members and hydrogen necessary to reduce more carbon dioxide. Bosch technology can achieve complete oxygen loop closure, but has many undesirable factors that result in a high energy, mass, and volume system. Finding a different catalyst with an equal reaction rate at lower temperatures with less catalyst mass and longer lifespan would make a Bosch flight system more feasible. Developmental testing of alternative catalysts for the Bosch has been performed using the Horizontal Bosch Test Stand. Nickel foam, nickel shavings, and cobalt shavings were tested at 500°C and compared to the original catalyst, steel wool. This paper presents data and analysis on the performance of each catalyst tested at comparable temperatures and recycle flow rates.

## Nomenclature

<i>BCaTS</i>	=	Bosch Catalyst Test Stand
<i>CO<sub>2</sub></i>	=	Carbon dioxide
<i>Co-Sh</i>	=	Cobalt Shavings
<i>CRA</i>	=	Carbon Dioxide Reduction Assembly
<i>ECLSS</i>	=	Environmental Control and Life Support Systems
<i>ISS</i>	=	International Space Station
<i>LEO</i>	=	Low Earth Orbit
<i>μGC</i>	=	Micro Gas Chromatograph
<i>MSFC</i>	=	Marshall Space Flight Center
<i>Ni-Sh</i>	=	Nickel Shavings
<i>NiF</i>	=	Nickel Foam
<i>SW-S</i>	=	Shredded Steel Wool

## I. Introduction

TO make long term space missions outside of Low Earth Orbit (LEO) possible, recovery of oxygen from metabolic carbon dioxide (CO<sub>2</sub>) is necessary. The current air revitalization technology onboard the International Space Station (ISS) is the Carbon Dioxide Reduction Assembly (CRA). This system uses a Sabatier reactor to reduce carbon dioxide with hydrogen, thereby forming water and methane (CH<sub>4</sub>) as shown below.

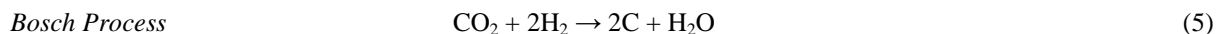
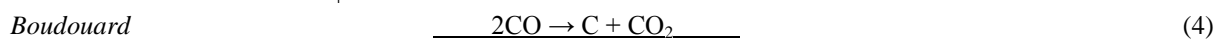
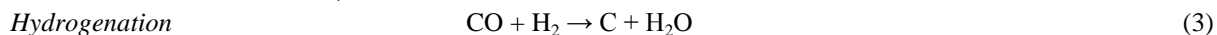


Product water is then fed to the Oxygen Generation Assembly where it is electrolyzed into oxygen for the crew, and hydrogen to be fed back to the CRA. Though the Sabatier can successfully reduce metabolic carbon dioxide, the

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reduction is not complete due to inadequate hydrogen lost in the form of methane. This makes resupply necessary, which is undesirable for long term space missions due to high launch costs and limited available space onboard. Bosch, a competitor of the Sabatier system, can reduce carbon dioxide with no loss of oxygen or hydrogen. First developed during the 1960's, the Bosch reactor traditionally reduces carbon dioxide with hydrogen over a steel wool catalyst to produce water and solid carbon by the following set of reactions.



This completely closes the oxygen and hydrogen loops, dramatically reducing the required amount of water and oxygen resupply.<sup>1</sup> When the air revitalization system for the ISS was being chosen, Bosch and Sabatier technologies directly competed against one another. Sabatier was the resounding victor for multiple reasons. First, full loop closure was deemed unnecessary for missions in LEO where resupply of water was easily accomplished. Additionally, the Sabatier is a much smaller mass, volume and energy system than the current Bosch, making it the best choice for carbon dioxide reduction for ISS.<sup>1</sup>

Many factors contribute to make the current Bosch system undesirable for future missions. Reactions over steel wool traditionally take place at temperatures around 650°C. Temperatures this high require significant power to maintain. The steel wool catalyst must be replaced often due to solid carbon fouling the catalyst and causing significant pressure drop. Replacing catalyst would prove particularly difficult in microgravity and could lead to possible contamination on board. If the system was to be flown in its current design, it would be large to accommodate the appropriate amount of catalyst to support a crew; unused replacement catalyst would have to be carried on board or resupplied; and old catalyst would need to be disposed of or stored. This would increase the volume of the reactor and storage volume needed to maintain the Bosch. Efficiency of the current Bosch reactor is low, and a large recycle stream must be used to reduce the necessary amount of carbon dioxide. All of these factors lead to a large mass, volume, and energy system undesirable for space flight.<sup>2</sup>

In an attempt to improve the Bosch system by finding a replacement for traditional steel wool, alternative catalysts were tested. Significant testing of the Boudouard and RWGS reactions was performed using several catalysts.<sup>3</sup> It is possible that one of these alternative catalysts could perform the Bosch process with the same efficiency as traditional steel wool but at a lower temperature and with a longer life. This could greatly improve the current mass, volume and energy requirements of the Bosch system making it the most advanced option for future long term space flights. This paper describes the Full Scale Alternative Catalyst testing performed with the Horizontal-Bosch (H-Bosch), including the methods used for testing and a discussion of all results.

## II. Hardware Description

Testing was performed using the H-Bosch located at Marshall Space Flight Center's (MSFC) Environmental Control and Life Support Systems (ECLSS) developmental facility. The H-Bosch, shown in Figure 1, was first developed by Life Systems Inc. and consists of several components. These include two reactor housings, a condensing heat exchanger, heaters, and items for monitoring and controlling reaction variables such as thermocouples, flow controllers, and pressure transducers. Due to damage inflicted prior to testing of the secondary reactor, only one reactor housing was used for this test. The reactor housing consists of two heaters: a sheathed core heater running axially approximately three fourths of the length of the reactor and a heat wrap placed on the outside. A coiled tube in tube heat exchanger surrounds the outside of the reactor.

A removable cartridge for holding catalyst is placed inside the reactor and is held in place by a face plate secured with a v-clamp.



Figure 1. Open H-Bosch reactor.

Reaction gases are fed into the reactor via a feed tube located around the core heater. Gas flows radially through the catalyst cartridge and linearly down the reactor away from the entrance point. Gas exits the reactor through a distributor at the base of the feed tube. The effluent gas then passes through the condensing heat exchanger where any product water is condensed and collected. The remaining stream passes through a compressor and is recycled back to the reactor.

The system is controlled via a LabVIEW-based custom interface (National Instruments, Houston, TX). The control system allows for the manipulation of flow controllers and heaters. It also shows and collects data from thermocouples, pressure transducers, and similar devices. An Agilent Technologies micro gas chromatograph ( $\mu$ GC) (Santa Clara, CA) monitors gas composition. Different points throughout the system can be chosen for sampling by using a multipoint valve manufactured by Valco Instruments Company (Houston, TX). The  $\mu$ GC is programmed to continuously sample the gas stream with three and a half minutes between each sample. Once a sample is completed, composition information is immediately sent to the primary control system, allowing the controller to vary gas feed rates based on stream composition.

### III. Methods

Four catalyst were chosen for FSACT, nickel shavings (Ni-Sh), nickel foam (NiF), cobalt shaving (Co-Sh), and base line shredded steel wool (SW-S) for comparison. Due to different densities and amounts of supplied catalyst, an approximate volume of 3.38L was targeted for each test. Actual masses used are shown in Table 1.

Insulation must line the cartridge to contain any loose catalyst or solid carbon particles. Two sheets of  $\frac{1}{2}$ " Fiberfrax (Niagara Falls, NY) Durablanket® S insulation were placed at the top and bottom of the cartridge, and a single sheet of  $\frac{1}{2}$ " insulation was wrapped around the center mesh and the outer mesh wall. At the top of the cartridge, a small hole was made in both insulation sheets so that the thermocouple well could penetrate into the center of the cartridge. This arrangement of insulation was used in each test.



**Figure 2. Center mesh piece from catalyst cartridge with part of NiF packing.**

throughout the empty cartridge volume and placed in the reactor without any further alterations to cartridge contents. The cobalt shavings were distributed through the volume of the reactor by rolling them in layers of insulation around the center mesh piece (Figure 3). The roll of insulation and Co-Sh catalyst were then inserted into the cartridge and sealed.

Steel wool was purchased from Global Materials Technologies (Buffalo Grove, IL). The

**Table 1. Mass of Catalyst Used**

Catalyst	Abbreviation	Mass (g)
Nickel Shavings	Ni-Sh	711
Nickel Foam	NiF	287
Cobalt Shavings	Co-S	110
Shredded Steel Wool	SW-S	150

Nickel foam sheets were

purchased from Novamet Specialty Products Corporation (Wyckoff, NJ). For packing, the foam was cut into 1.5" by 6.5" strips and 6.5" diameter disks. The strips were folded, and both disk and strips were packed alternately to produce six layers of alternating foam pieces show in Figure 2.

Both nickel and cobalt shavings were prepared from 99% pure metal rods from ESPI Metals (Ashland, OR). Shavings were prepared from the rods and are approximately 0.127 mm (0.005") thick. For packing, the Ni-Sh catalyst was distributed



**Figure 3. Layers of Co-Sh on insulation being placed around cartridge center.**

steel wool was supplied as a rolled bundle with strands with an average diameter of 25  $\mu\text{m}$  and length of 61 cm. Pieces were taken off the roll and cut into approximate  $\frac{1}{2}$ ” squares. Before packing, the steel wool was pretreated by cleansing with 3% hydrochloric acid, rinsing with de-ionized water, and then baking for 45 minutes at 207°C. This process was used to deoxidize the catalyst. After pretreatment, catalyst was immediately packed in to the cartridge and placed into the reactor. The reactor was then purged with nitrogen, and the catalyst was left overnight.

### A. H-Bosch Operation

The Full Scale Alternative Catalyst Testing was conducted at 500°C. A system pressure of 28 psia was maintained during testing, and a 2:1 ratio of hydrogen to carbon dioxide was targeted in the system. Before testing could begin, the reactor was pre-heated containing only carbon dioxide. Once the reactor reached 250°C, hydrogen was introduced by purging to depressurize the system and re-pressurizing with hydrogen. The start of each test trial was considered the moment when the reactor reached the desired operating temperature. Reactor pressure and recycle stream ratio were controlled by re-pressurizing the system with the feed gases when the pressure dropped due to either reactions taking place or reactor leakage. Recycle ratios of 70, 50, and 30 SLPM were tested in varying orders within each trial for one and a half hours as seen in Table 2. Once testing was completed the reactor was allowed to cool overnight. Once cooled, the catalyst cartridge was removed from the reactor and the catalyst inside examined, documented, and stored.

Table 2. Test Schedule Example

Catalyst	Trial	Run	Recycle Flow Rate (SLPM)
Ni-Sh	1	1	30
		2	50
		3	70
	2	1	50
		2	70
		3	30
	3	1	70
		2	30
		3	50

## IV. Results and Discussion

Four catalysts were evaluated in Full Scale Alternative Catalyst Testing. Traditional shredded steel wool, cobalt shavings, nickel shavings, and nickel foam were tested under comparable conditions. Data was collected throughout the test, including stream composition data, gas feed amounts, and water production, as well as others.

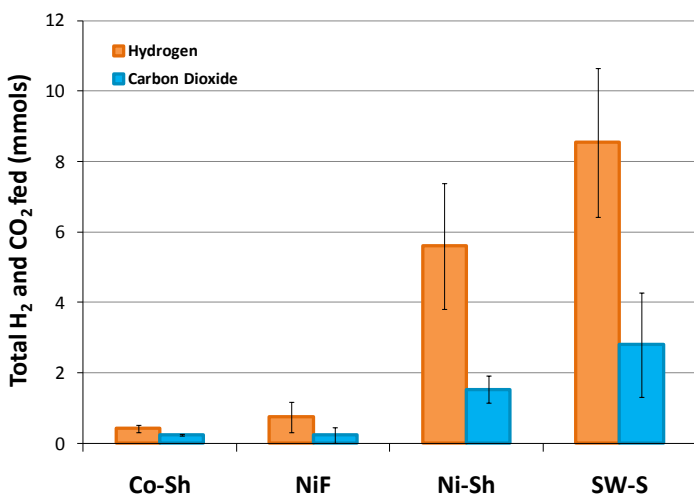


Figure 4. Average total hydrogen and carbon dioxide fed to system for each catalysts' test trials.

general, more hydrogen is fed to the system than carbon dioxide to maintain the 2:1 ratio. This is because more moles of hydrogen are needed to carry out the reactions, which is easily seen in the balanced equations 1-5 in the introduction.

### A. Total Reactants Fed

The total mass of hydrogen and carbon dioxide fed to the system during each test was evaluated from recorded flow rates. As these reactants are used up, pressure drops within the system and more hydrogen and carbon dioxide must be introduced. Therefore, the amount of reactant fed is directly related to products made. Figure 4 shows the average total mass of hydrogen and carbon dioxide fed per trial for each catalyst tested. SW-S used the most reactants followed by Ni-Sh. Co-Sh and NiF used a comparable amount of hydrogen and carbon dioxide. More reactants fed should correlate to the activity of the catalysts, so it would be expected from these results to see more water for SW-S and Ni-Sh than the other two catalysts. It can also be seen that, in

### B. Recycle Flow Rate Compositions

The composition of the recycle stream was monitored by the  $\mu$ GC throughout testing. Figure 5 shows the average recycle flow rate composition for each test catalyst. Hydrogen, carbon dioxide, carbon monoxide, and methane are shown, respectively. Co-Sh and NiF show some carbon monoxide and little to no methane. This indicates the desired reverse water-gas shift reaction is selectively occurring over the Sabatier reaction. Both Ni-Sh and SW-S showed carbon monoxide formation, but also a large amount of methane build-up. For methane to be seen in this amount for these catalysts, the Sabatier reaction was occurring more often than desired for the Bosch system. This methane formation would also cause more hydrogen to be fed to maintain the 2:1 ratio, as hydrogen would be lost to the methane product.

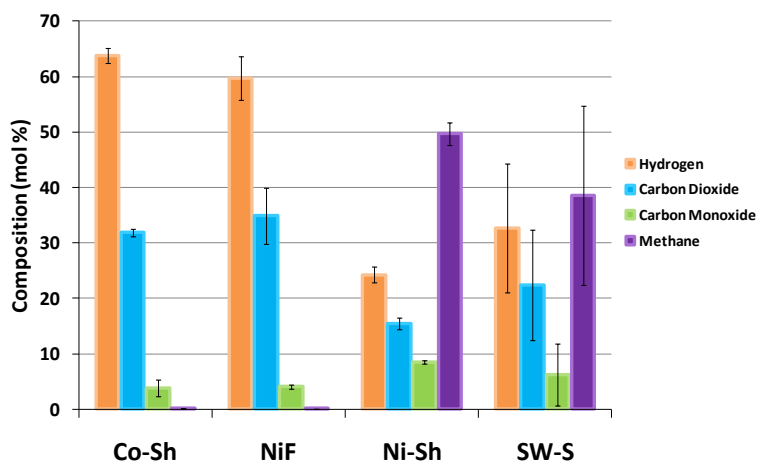


Figure 5. Average recycle stream percent composition for each catalyst.

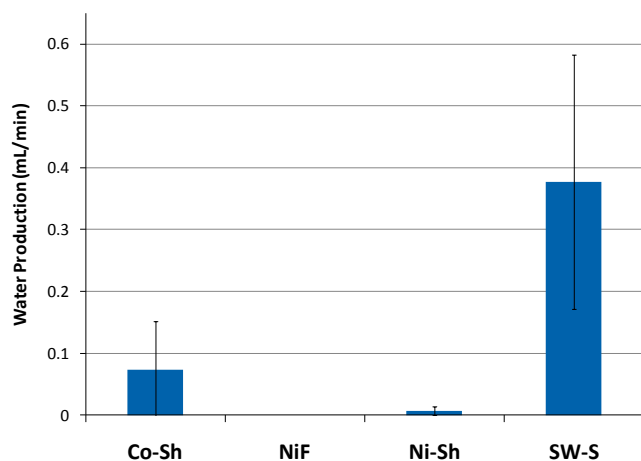


Figure 6. Average water production rates for each catalyst.

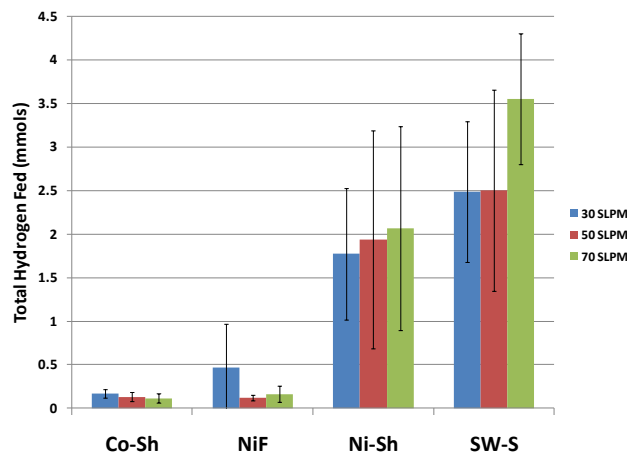


Figure 7. Average total hydrogen fed for each flow rate and catalyst.

### C. Water Production Rate

Water was collected between each recycle flow rate run. Due to the small amounts collected for Co-Sh, NiF, and Ni-Sh, water could only be collected at the end of the trial. Figure 6 shows the average water production rate for each catalyst. Water is reported as a rate since each test varied with respect to total test length. SW-S produced the most water per minute followed by Co-Sh. Ni-Sh produced minimal water, and NiF produced no detectable water.

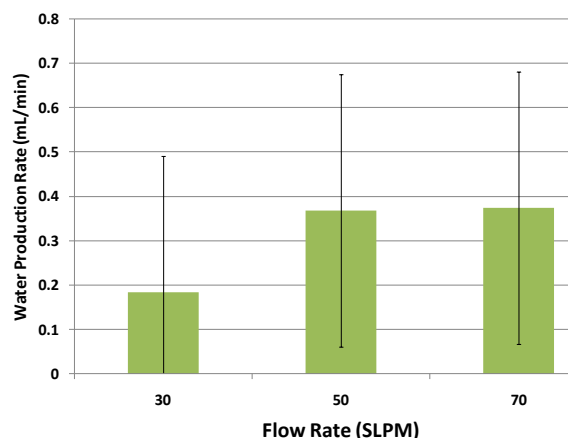
### D. Recycle Flow Rate Effects

No data showed any statistical difference between flow rates for any catalyst. Figure 7 shows the average feed hydrogen for each flow rate and for each catalyst. Error bars greatly overlap indicating no statistical difference when comparing flow rates and no trend is apparent. The same can be said for the water production rate for SW-S. SW-S produced enough water for water samples to be taken between runs. Figure BBB shows the average water production rate for each flow rate on SW-S. Once again no statistical difference can be seen between flow rates. This shows that residence time had no effect on catalyst activity during testing.

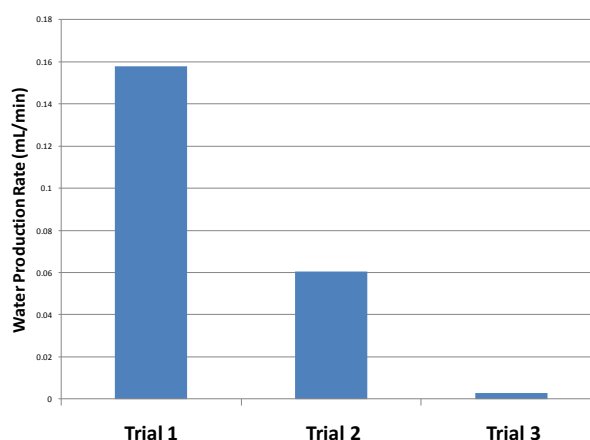
### E. Overall Data Comparison

While comparing the results, some performance-based conclusions are contradictory. The Ni-Sh catalyst was fed more reactants than Co-Sh or NiF, but had less water collected than Co-Sh and only marginally more water was produced than NiF. Co-Sh





**Figure 9. Shredded steel wool water production rates for each flow rate.**



**Figure 8. Cobalt shavings water production rate for each trial.**

was fed the least but produced the most water out of these three. Several test factors could have led to these results. The Co-Sh catalyst was tested after an extended break in testing. It is highly probable that during this time period water from the atmosphere collected in the H-Bosch system piping. This theory is also strengthened by the data in Figure 9 showing each trial's water production rate in the order performed. The first trial produced a staggering amount of water in comparison to the following two. Also during the testing of Co-Sh the tank was "tipped" so that all the water below the exit valve could also be collected. This emptying process was not used for either NiF or Ni-Sh, meaning that more water was possibly produced in both tests but was not collected.

## V. Conclusion

It has been shown here that steel wool catalyst is still the best option for a single reactor Bosch, even at reduced temperature. None of the alternative catalysts performed within the same margins as steel wool, producing less than half of the water than the steel wool catalyst. The future of the Bosch is moving away from a single reactor to reactors in series. Already in use is the Bosch Catalyst Test Stand (BCaTS) to test this concept. Bosch reactors in series can take advantage of the different selectivities for each reaction, including temperature and catalyst. This could vastly improve the single pass efficiency for the Bosch reaction. Also, a planned redesign of the Bosch taking advantage of reactors in series is planned for FY12.

## Acknowledgments

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