The Technology and Future of In-Situ Resource Utilization (ISRU)
The University of Central Florida

Mars Atmospheric Capture [and Processing]

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February 27, 2017
• Martian resources
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Martian Resources

- Atmosphere of Mars
  - 95.9% CO₂
  - 2% Ar, 1.9% N₂
  - <1% pressure of Earth’s atmosphere (~7 mbar)

- Significant Amounts of Water in the Top 1-Meter of Regolith
  - Water ice caps at the poles
  - ~2% at least everywhere else
  - ~10% even at equatorial regions
  - Curiosity rover ground truth:
    - 1.5-3% water in surface regolith (SAM)
    - Average 2.9% water (DAN), up to 7% in top 60 cm of regolith in some locations-seasonal variation
  - Transient liquid water at night in the top 5 cm of regolith

Regolith volatiles analysis by SAM
Orbital estimates of water in top 1-m of regolith via slow neutrons

Water content of regolith vs. distance by DAN on Curiosity Rover
Introduction

• In 1976, Robert Ash at Old Dominion U. initiated idea of Mars ISRU using CO$_2$ from the atmosphere & water from regolith
• Concept: make methane and oxygen via the Sabatier reaction and water electrolysis:
  – CO$_2$ + 4 H$_2$ $\rightarrow$ CH$_4$ + 2 H$_2$O
  – 2 H$_2$O $\rightarrow$ 2 H$_2$ + O$_2$
  – Freezing using liquid methane for CO$_2$ collection
• First demonstrated by Zubrin, Clark et al. at Lockheed Martin Astronautics in early ‘90s, but w/H$_2$ from Earth
  – Used zeolite sorption/desorption for CO$_2$ collection
  – Leverage: 12:1, but 10.3:1 after discarding excess CH$_4$
  – Supplement w/RWGS or CO$_2$ electrolysis $\rightarrow$ O$_2$
  – Raises leverage to 18:1
• Main issue w/imported H$_2$: size of H$_2$ tanks
  – Low density/high boil-off
  – Too large for practical shrouds & heat shields

Why Mars ISRU?

- “Gear ratio” = mass in LEO/landed mass on Mars surface = ~10
- 10 kg IMLEO/1 kg on Mars surface
- Example: MAV (Mars Ascent Vehicle) = ~6 tons dry mass
- Propellant
  - 7 tons CH$_4$ + 23 tons O$_2$
  - 5 tons life support O$_2$
  - Total = 35 tons
- IMLEO ~ 300 tons or ~several HLLVs (saves $billions)
- Mars atmosphere/water ISRU system ~1.7 tons (regolith)
  - ~2.2 tons w/life support water product
- Reduces landing mass by ~30 tons or ~60%!
- Power ~40 kW, but also needed for crewed phase
  - Not counted against ISRU system mass

Dust Removal

• Martian atmosphere contains significant amounts of dust, especially during dust storms
• Can interfere with valves, catalysts, pumps, etc.
• NASA developing three approaches
  – Cyclone separator and HEPA filters (Juan Agui at GRC)
  – Electrostatic dust removal (Carlos Calle at KSC)
Adsorption/Desorption - MIP

- MIP: Mars In-situ Propellant Production Precursor planned for the canceled Mars Surveyor 2001 Lander
- Led by David Kaplan at JSC
- Mass = 8.5 kg, Power (avg) = 16.0 W
- 40 cm x 24 cm x 25 cm box
- Oxygen Generator Assembly (OGA) Electrolyzes CO₂ to oxygen (~10 g/h) and carbon monoxide
- Used zeolite sorption/desorption (13X) (MAAC from JPL) with night/day cycle (200 K – 450 K)
  - 4.5 g CO₂/6 h at 0.58 atm pressure
- Designed to run for 300 Mars days (sols).
The end-to-end test started with the adsorption bed removing carbon dioxide from the simulated Mars atmosphere. The bed was then heated to transfer CO2 to the Sabatier reactor with H2 gas which was catalytically converted to CH4 and H2O. The H2O vapor is condensed and delivered to the H2O electrolysis unit where it is electrolyzed into H2, which is recycled back to the Sabatier reactor, and O2, which is dried and delivered to the liquefaction and storage system. The CH4 stream from the Sabatier reactor contains residual H2 since the process is run H2 rich to maximize the conversion of CO2. The CH4 stream is passed through an electrochemical membrane to recover the H2 (and a good portion of the water) and recycle it back to the Sabatier reactor. Finally, the CH4 is passed through a drier and then routed to the liquefaction and storage system. In initial tests, the methane was simply vented overboard, since only one cryocooler was available for the system (used for O2 liquefaction). The test was run under simulated Mars conditions for 9 days, and test results were favorable, with almost complete conversion of the CO2, maximized usage of the H2, and smooth operation of the integrated breadboard. Figure 11 depicts the end-to-end Mars ISRU test article and the Mars environment simulation chamber at JSC.
Freezing/Sublimation

- **CO₂ Freezers Look Promising**
- CO₂ freezers tested by Pioneer Astronautics, Lockheed-Martin (two scales), and KSC
- Results: ~20, 13, 80, and 100 g/hr using lab-scale systems
  - (equiv. 5-32 g/hr CH₄)
- Thickness up to 1 cm
- N₂/Ar was not measured or purified
- CO₂ self-pressurizes via sublimation
- Don Rapp (JPL) estimated a CO₂ freezer for 0.5 kg/hr needs ~1/3 the power and 11% the mass of a compression pump/membrane CO₂ purifier

**TRL 3-6**

Pioneer MACDOF (LN₂ Chilling)

KSC “Ferris Wheel” Cold Head Testing

Lockheed Cryocooler Freezer

0.9 Watt-h/g CO₂
Direct Compression/Liquefaction

- CO₂ Liquefaction and Collection of Other Gases
- Compress very large volumes of the atmosphere to the high pressures required to liquefy CO₂
- Geared toward larger scale operations: settlements
- Requires very high power source - nuclear reactor
- Not appropriate for Mars Sample Return or early human exploration

Mars Atmosphere Resource Recovery System (MARRS) – NIAC Study

TRL 1-2

Direct Compression & Processing

- ISRU processes (SOE, RWGS, Sabatier) tolerate lower purity CO\textsubscript{2}
- Pioneer Astronautics tested combined RWGS-Sabatier process with CO\textsubscript{2}/N\textsubscript{2}/Ar for 5 continuous days without degradation of catalyst
  - 1 kg/day of 3.5 O\textsubscript{2} to 1 CH\textsubscript{4}
  - Power = 893 W (678 W optimized)
- N\textsubscript{2} and Ar not separated, but removed during condensation or cryodistillation of products
- Gas separation downstream from CO\textsubscript{2} reduction process may be easier and still provide useful buffer gases
- Two-stage COTS mechanical compression required 242 W (plus chiller), and may require more power than freezing, but was claimed to be less complex
- Mass comparison needs to be done

TRL 4

Pioneer IMISPPS
46 cm x 41 cm x 94 cm; 115 kg (54 kg optimized)
MOXIE – Example of Direct Compression
MOXIE is the Mars OXygen In situ resource utilization Experiment on the Mars 2020 Rover.

Led by Dr. Michael Hecht, MIT/Haystack Obs.

Electrolyzes CO₂ to oxygen (~10 g/h) and carbon monoxide.

Initially specified a small cryocooler.

Switched to a small scroll pump from Air Squared (Broomfield, CO).

N₂ and Ar are not separated from feed, but vented with CO.

Compressor Requirements:

- A mass flow rate of at least 100 grams per hour
- An outlet pressure of at least 760 Torr, with 7 Torr inlet pressure
- A compressor and motor not exceeding 175 mm in length
- Full compatibility with the working fluid: 95% CO₂, 3% N₂, and 2% Ar
- A total mass of compressor, motor, and controller not exceeding 1.8 kg
- The ability to function in the Martian environment
- Further improvements in size, weight, and efficiency throughout development

CO₂ Capture with Membranes

- Evaluated 28 other membrane materials
  - Top 10 identified
- Mainly polymeric-based: poly-acetate, polyimides, polyamides, poly-sulfone, polycarbonates, and polyethylene, plus zeolite membranes
- Selectivity and permeability are inversely related
- Pressurization is required (1-10 atm)
- Polyacetylene and polydimethylsiloxane have the highest permeability
- Trades are needed for selectivity vs permeability and power to compress the CO₂ for separation
- Synthesis required in some cases

Distribution of membrane permeability. Note: For clarity, the top two ranked materials are not included in this graph (1 barrer = 7.5 x 10⁻¹⁴ (cm³(STP)-cm)/(cm²-s-Pa)).

TRL 2-3
Air Products Prism modules have been tested by LMA, Pioneer Astronautics and KSC. KSC results are good with minor H2 losses (0.26% average).

<table>
<thead>
<tr>
<th>Stream</th>
<th>Calculated H2 (slpm)</th>
<th>Measured H2 (slpm)</th>
<th>Calculated CO2 (slpm)</th>
<th>Measured CO2 (slpm)</th>
<th>Calculated CO (slpm)</th>
<th>Measured CO (slpm)</th>
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Ionic Liquids for CO$_2$ Adsorption/Electrolysis

Typical cations and anions for ILs

Ionic Liquids are organic, ionic compounds that are liquid at or near room temperatures
Potential Benefits for ISRU

Current Mars Bipropellant Production Process Diagram

- Advantages of IL capture/electrolysis:
  - No high temperature processing of CO₂
  - One less pump and no cryocoolers
  - Four fewer major process steps
  - Estimated ~50% less mass and ~25% less power
CO₂ Uptake at Low Partial Vacuum ~50% Mole Fraction at ~10 mbar

“CO₂ absorption capacity in (a) [emim][2-CNPyr], (b) [emim][4-Triaz], (c) [emim][3-Triaz], and (d) [emim][Tetz] at 22 °C. The CO₂ solubility in [P₆₆₆₁₄]+ counterparts from ref 10 are also shown for comparison.” (Brennecke, 2014)
## Summary of KSC Results
(Underlined ILs = Candidates)

<table>
<thead>
<tr>
<th>Ionic Liquid</th>
<th>CO$_2$ Capacity, wt.% (R.T., 1 atm, dry)</th>
<th>Electrochemical Window, V</th>
<th>Conductivity with CO$_2$ (mS/cm, 40°C)</th>
<th>Compatible with Cu</th>
<th>Other Issues</th>
<th>Tested Solubility of Water, v/v%</th>
<th>Methane Production Rate</th>
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<td>[BMIM][TFSI]</td>
<td>0.46</td>
<td>2.1</td>
<td>No</td>
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<td></td>
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<td>[BMIM][PF$_6$]</td>
<td>0.50</td>
<td>2.4</td>
<td>Yes</td>
<td>Precipitate, Cu darkened</td>
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<td></td>
<td>0</td>
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<td>[BMIM][BF$_4$]</td>
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<td></td>
<td></td>
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<td>[HMIM][B(CN)$_4$]</td>
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<td>AZ-1</td>
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<td>15.6</td>
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<td>Slow color change</td>
<td>Precipitate</td>
<td>5</td>
<td>Possible CH$_4$ and CO (TiO$_2$ only)</td>
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</table>
Ionic Liquids for CO$_2$ - Summary

• In general, the benefits of ILs are:
  – Low volatility
  – Wide range of regeneration temperatures
  – Less energy to regenerate
  – Potentially lower corrosion
  – High temperature stability
  – Ability to tune performance

• Some major challenges for ILs that still need to be addressed:
  – Limited commercial availability
  – Limited understanding of the reaction mechanisms and kinetics

• Some major challenges for IL membranes that still need to be addressed:
  – Improvements in membrane fabrication (in general)
  – Improvements in large scale membrane fabrication
  – Improvements in producing defect free coating for the membranes
Buffer Gas Separation

- **COTS Membrane Modules Are Adequate**
- Parrish (KSC, 2002) studied several commercial membranes:
  - Permea Prism® Alpha Separators PPA-20.
  - Neomecs GT #020101.
  - Enerfex SS.
  - Enerfex SSP-M100C Membrane sheet.
- **Temperatures = -45°C to +30°C.**
- **Variety of pressures.**
- Designed a system that would operate at -44°C and 780 mm Hg (1.03 atm)
- **Feed = 30% CO₂, 26% Ar, and 40% N₂.**
- **Predicted product = 6 lpm, 600 ppm CO₂, 38% Ar and 62% N₂.**
- **47% recovery of the feed.**
- **Work is needed on Ar/N₂ separation.**
  - Ar leads to potential bends issue.

Membrane purification of feed from the capture of CO₂ (Parrish, 2002)

**TRL 3-4**

Microchannel Technologies

- Microchannel reactors offer:
  - Better temperature control of the catalyst bed
  - Reduce temperature gradients and localized “hot spots”
  - Prevent sintering of a packed bed catalyst
  - Large mass savings over the traditional packed bed reactor design,
  - Penalty of increased pressure drop and increased probability of complete catalyst deactivation.
- Potentially improved CO$_2$ absorption for concentration
  - Lower mass, volume, and power
- Further development is justified

TRL 3
MARCO POLO/
Mars ISRU Pathfinder

- **ISPP: In Situ Propellant Production**
  - Demonstrate production of Mars Sample Return propellant
  - Reduce risk for human Mars missions

- **MARCO POLO - Mars Atmosphere and Regolith COLlector/PrOcessor for Lander Operations**

- The Mars Atmospheric Processing Module (APM)
  - Mars CO₂ Freezer Subsystem
  - Sabatier (Methanation) Subsystem

- Collect, purify, and pressurize CO₂
- Convert CO₂ into methane (CH₄) and water with H₂
- Other modules mine regolith, extract water from regolith, purify the water, electrolyze it to H₂ and O₂, send the H₂ to the Sabatier Subsystem, and liquefy/store the CH₄ and O₂
- Test in a Mars analog (field demo)
What is MARCO POLO?

• First generation integrated Mars soil and atmospheric processing system with mission relevant direct current power
  – 10 KW Fuel Cell for 14 hrs of daytime operations
  – 1KW Fuel Cell for 10 hrs of night time operations

• Demonstrates closed loop power production via the combination of a fuel cell and electrolyzer.
  – The water we make and electrolyze during the day provides the consumables for the 1KW Fuel Cell that night

• Planned for remote and autonomous operations
Lander Design Concept

Atmo Processing Module:
- CO2 capture from Mixed Mars atmosphere (KSC)
- Sabatier converts H2 and CO2 into Methane and water (KSC/JSC)

Water Processing Module: (JSC)
- Currently can process 520g/hr of water (max 694 g/hr)

Liquefaction Module: (TBD)
- Common bulkhead tank for Methane and Oxygen liquid storage

C&DH/PDU Module: (JSC)
- Central executive S/W
- Power distribution

Soil Processing Module: (JSC)
- Soil Hopper handles 30kg (KSC)
- Soil dryer uses CO2 sweep gas and 500 deg C to extract water

Water Cleanup Module: (KSC)
- Cleans water prior to electrolysis
- Provides clean water storage

Excavator: (KSC)
- RASSOR 2.0 Rover
- Provides feed to Soil Dryer

1KW Fuel Cell and consumable storage (JSC & GRC)
- Uses metal hydride for H2 storage due to availability
- 1KW No Flow Through FC (GRC)
- 10KW FC not shown (JSC)

3m x 3m octagonal lander deck
Existing Hardware

KSC: Mockup Lander w/Hopper, Mock Oven, and APM Simulator in Regolith Bin

KSC: RASSOR 2.0
Excavator Rover

KSC: Common Bulkhead Cryotank

JSC: Soil Processing Module

KSC/JSC: Water Cleanup Module

JSC: Water Processing Module (Electrolyzer)

KSC: Atmospheric Processing Module
APM Goals/Requirements

- Collect and purify 88 g CO₂/h (>99%)
  - From simulated Martian atmosphere
  - 10 mbar; 95.4% CO₂, 3% N₂, 1.6% Ar
- Supply 88 g CO₂/h at 50 psia to the Sabatier reactor
- Convert CO₂ to 32 g CH₄/h and 72 g H₂O/h
- Operate autonomously for up to 14 h/day
- Minimize mass and power
- Fit within specified area and volume
  - 9,000 cm² pentagon
  - 10,000 cm² rectangle for easier lab operations
  - 44 inches tall (112 cm, same as Water Processing Module)
- Support MARCO POLO production goals of 0.032 kg CH₄/h and 0.128 kg O₂/day (50% of O₂) for a total of 2.22 kg propellant/14 h day
- Sufficient for a Mars Sample Return Mission
- ~13% of full-scale O₂ production goal for human Mars Missions (1 kg O₂/h/module x 3 modules = 3 kg O₂/h), i.e. 1/8th scale
Atmospheric Processing Module Design

- Sabatier Reactor
- Methane Dryer (Future)
- CO\textsubscript{2} ballast tanks not shown
- Chiller
- CO\textsubscript{2} Freezers
- Electro-chemical Methane Separator
- Vacuum Pump

[Replaced by Recycle Pump and Membrane Module]
Atmospheric Processing Operations

- Mars Mix
  - Ballast tank
  - Ballast tank
  - CO₂ freezer
  - CO₂ freezer

- Sabatier Reactor (<600 deg C)
  - 88 g/hr CO₂ at >50 PSI

- CH₄/H₂ Separator
  - 2 g/hr H₂
  - 72 g/hr H₂O
  - 32 g/hr CH₄
  - 2 g/hr H₂

- Condenser
  - Water Cleanup Module
  - H₂O
  - CH₄

- CH₄ Dryer
  - CH₄ storage

- Electrolysis Stacks
  - 16 g/hr H₂

- Water Processing Module

- 95% CO₂, 3% N₂, 2% Ar at 10.8 mbar
CO₂ Freezer – Final Design

Mars Atmosphere
95% CO₂, 3% N₂, 2% Ar
~700 psig max

- 15 psig
- CO₂ Pump
- CO₂ Ballast Tanks
- Pressure Equalizer Valves
- Vent to Atmosphere/Hood
- Pressure Transducers, etc.
Design of KSC Sabatier Reactor

- 30 cm long stainless steel tube w/ 2.54 cm OD and 0.21 cm wall thickness
- Single-pass conversion = 90% @ 88 g CO₂/h + 3.5:1 H₂/CO₂
- Based on Pioneer Astronautics design for steam oxidation of trash to methane
- 1.5 h integrated test with CO₂ Freezers and recycling system showed 100% conversion to pure CH₄
Atmospheric Processing Module

- Sabatier Reactor
- Membrane Module
- Recycle Pump
- CO₂ Storage Tanks
- Avionics
- CO₂ Freezeers
- Copper Heat Exchanger
- CO₂ Freezers and Chiller
3D Model of the APM Major Components

Major component mass = 154 kg
Water Cleanup Module (KSC)

- Tested with Water Processing Module at JSC
- Used to recycle fuel cell water from the MMSEV to $\text{H}_2$ and $\text{O}_2$
- MMSEV = Multi Mission Space Exploration Vehicle

Ion exchange resin instead of membrane separator included in the final version
Van Townsend (KSC/ESC) with MARCO POLO lander and Soil Processing Module (under construction)

RASSOR (Regolith Advanced Surface Systems Operations Robot) will feed the hopper (2 x 40 kg drums, 25 cm/s, 60% on duty time)
**CO₂ Freezer Testing**

- **Avg. Capture Rate** = 99±3 g/hr at 1.2 SLPM (1.4 hr test)
- **Avg. Sublimation Rate** = 95±8 g/hr (1.4 hr test)
- **Avg. Capture Fraction** = 79±3%
- Exceeds 88 g/hr requirement
- Better performance than test stand!

**Graph Details**

- **Mars Atmosphere Simulant Flow Rate, SLPM**
- **% CO₂ Capture**
- **CO₂ Collection Rate, g/hr**
- **CO₂ Sublimation Rate in 1.4 hr, g/hr**
- 88 g/hr Requirement
Long-Duration Tests Were Successful

Due to pressure losses during manual draining of Sabatier water condenser

<table>
<thead>
<tr>
<th>Run No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sabatier Run Duration</td>
<td>7.0 h</td>
<td>7.0 h</td>
<td>7.0 h</td>
<td>7.0 h</td>
</tr>
<tr>
<td>Gas Composition</td>
<td>CO₂</td>
<td>CO₂</td>
<td>Mars Gas</td>
<td>Mars Gas</td>
</tr>
<tr>
<td>Average CO₂ Freezing Rate (Goal = 88 g/h)</td>
<td>102 g/h</td>
<td>100 g/h</td>
<td>102 g/h</td>
<td>99 g/h</td>
</tr>
<tr>
<td>Average Fraction of CO₂ Frozen</td>
<td>80%</td>
<td>70%</td>
<td>70%</td>
<td>73%</td>
</tr>
<tr>
<td>Average Cryocooler Power</td>
<td>139 W</td>
<td>150 W</td>
<td>158 W</td>
<td>138 W</td>
</tr>
<tr>
<td>Average “Lift” Needed to Freeze CO₂</td>
<td>0.19 W/g/h</td>
<td>0.21 W/g/h</td>
<td>0.22 W/g/h</td>
<td>0.20 W/g/h</td>
</tr>
<tr>
<td>Average CO₂ Supply Rate to Freezers</td>
<td>128 g/h</td>
<td>142 g/h</td>
<td>146 g/h</td>
<td>135 g/h</td>
</tr>
<tr>
<td>Average CH₄ Production Rate</td>
<td>32 g/h</td>
<td>32 g/h</td>
<td>32 g/h</td>
<td>32 g/h</td>
</tr>
<tr>
<td>Average CH₄ Purity</td>
<td>99.9%</td>
<td>99.9%</td>
<td>96.0%*</td>
<td>99.9%</td>
</tr>
<tr>
<td>Average H₂O Produced</td>
<td>67 g/h</td>
<td>69 g/h</td>
<td>64 g/h</td>
<td>70 g/h</td>
</tr>
<tr>
<td>Missing H₂O (vs. 72 g/h)</td>
<td>6.9%</td>
<td>4.2%</td>
<td>11.1%</td>
<td>2.8%</td>
</tr>
</tbody>
</table>

*Due to pressure losses during manual draining of Sabatier water condenser
Selected Results from Long-Duration Tests

CO₂ Freezer Cold Head Temperatures and Cryocooler Power Consumption during the Third Run of the 7-h Integrated Test Series

Sabatier Reactor Temperatures during the Second Run of the 7-Hour Integrated Test Series
Conclusions from the Long-Duration Tests

• **CO₂ Freezer Subsystem operates well**
  – Exceeds 88 g/h freezing and supply rate
  – Freezes ~70% of incoming CO₂
  – Provides valuable data for power to freeze CO₂ at Mars pressure
    • Averages 0.22 W/g CO₂ frozen = only 108% of theoretical
  – Contributes to Human Mars Mission ISRU system designs, e.g. 680 W lift for 3.1 kg CO₂/h

• **Sabatier Subsystem also operates well**
  – New reactor is efficient
  – Recycling system (membrane module + recycle pump) works well (but recycle pump failed recently)
  – Pure CH₄ obtained at expected rate
  – ~6% of water is missing (<1% of loss is in CH₄)
Recent Work and Current Status

- Additional integrated tests performed
- Faster and slower production rates tested
  - 1.0-1.6 SLPM feed to CO$_2$ Freezers (87-71% frozen; 4800-5400 J/g)
  - Sabatier works at 0.3 to 1.2 SLPM CO$_2$ (0.75 SLPM nominal, 550°C max T)
  - Some CO observed in CH$_4$ after higher flow rates (catalyst damaged)
- Better LabVIEW automation implemented (sequences)
- Performed “virtual” integrated MARCO POLO tests with other systems at KSC in May and September – Hardware integration planned in FY17
- Ran Sabatier Subsystem w/Mars Gas Simulant – Good results. But Ar & N2 in CH4
- Now modeling CO$_2$ Freezers and Sabatier for scaling up to full-scale versions
- Long Term Goal is to continue to refine ISRU technologies for potential robotic Mars missions using Mars Pathfinder (2026/28)
APM Video
Video of Virtual Integrated Test

https://www.youtube.com/watch?v=cRLvAeL3wdU
Challenges

• Trade Studies based on small-scale tests and modeling followed by testing of full-scale components and subsystems up to TRL 6
• Funding (as usual)
Future Direction
[Tony’s Wish List – Same As 2 Weeks Ago]

• NASA has restarted its ISRU Project
• NASA should fully embrace ISRU
• Integrated human exploration of the inner solar system
• Moon, asteroids, Mars moons, Mars
• Commercial development of key technologies in partnership w/NASA to reduce costs
  • i.e. shared funding
  • Habitats, ISRU, spacecraft, launch vehicles, etc.
• Asteroid mining
• Pick an architecture and stick with it for >>8 years