EFFICIENT MICROWAVE APPROACHES FOR EXTRACTING WATER FROM HYDRATED MINERALS

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VALUE OF WATER FOR ISRU OPERATIONS

- **Major source of Oxygen and Hydrogen via Electrolysis**
  - Electrolyzers can electrochemically dissociate water and separate products hydrogen and oxygen
  - PEM and SOX electrolyzers have successfully demonstrated the ability to electrochemically produce high quality hydrogen and oxygen
    - Each has their own strengths and weaknesses as applied to ISRU operations
- **Water must be of high purity**
  - Contaminants in water feedstock to electrolyzers will shorten life of electrode catalysts
  - Water cleanup is a constant companion to electrolyzer operations
- **Availability of water affects efficiency of all downstream operations**
  - Constant supply of water reduces the requirement for repetitive shutdown/startup operations
  - Storage of a clean water source aids in both human and propellant operational demands
    - Clean liquid water stored for feedstock to electrolyzers assists in entire system operations, including thermal and habitat management operations

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

FOUR GENERAL SOURCES OF WATER ON MARS HAVE BEEN PROPOSED(*) (HLS^2 based)

- **Subsurface glacial remanants**
  - Ice buried below surface overburden
- **Poly-hydrated Sulfate**
  - 40% gypsum, 3.0% allophane, 3.0% akaganeite, 3.0% smectite in sand substrate
- **Clay**
  - 40% smectite, 3.0% allophane, 3.0% akaganeite, 3.0% bassanite in sand substrate
- **Typical Regolith**
  - 23.5% basaltic glass, 3.0% allophane, 3.0% akaganeite, 3.0% bassanite, 3.0% smectite in sand substrate

[Gypsum - $CaSO_4 \cdot xH_2O$; Allophane - $Al_2O_3 \cdot (SiO_2)_{1.3\text{-}2} \cdot xH_2O$; Akaganeite - $Fe^{3+}O(OH,Cl)$; Smectite - $(Na,Ca)_{0.33}(Al,Mg)_2(Si_4O_{10})(OH)_2 \cdot xH_2O$ (mordenite for example); Bassanite – [Gypsum]]

(*) Mars Water In Situ Resource Utilization (ISRU) Planning (M-WIP) Study

CONSIDERATIONS AFFECTING WATER EXTRACTION FOR ISRU

- **Type of water**
  - Hydrated salts
  - Clay
  - Ice
  - Adsorbed water
- **Energy requirements**
  - Energy required to ‘prepare/collect’ material that is water source
  - Energy into solid material
    - Energy to release water
    - Energy to volatilize water
    - Energy for water to escape its ‘confines’, i.e., environment surrounding the volatile water
- **Rate of water recovery**
  - Energy into material (soil, ice, etc.)
  - Extraction of water from material
- **Co-release of contaminants**
  - Sulfur
  - Halides

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

ILLUSTRATION OF POTENTIAL WATER CONTAMINANTS

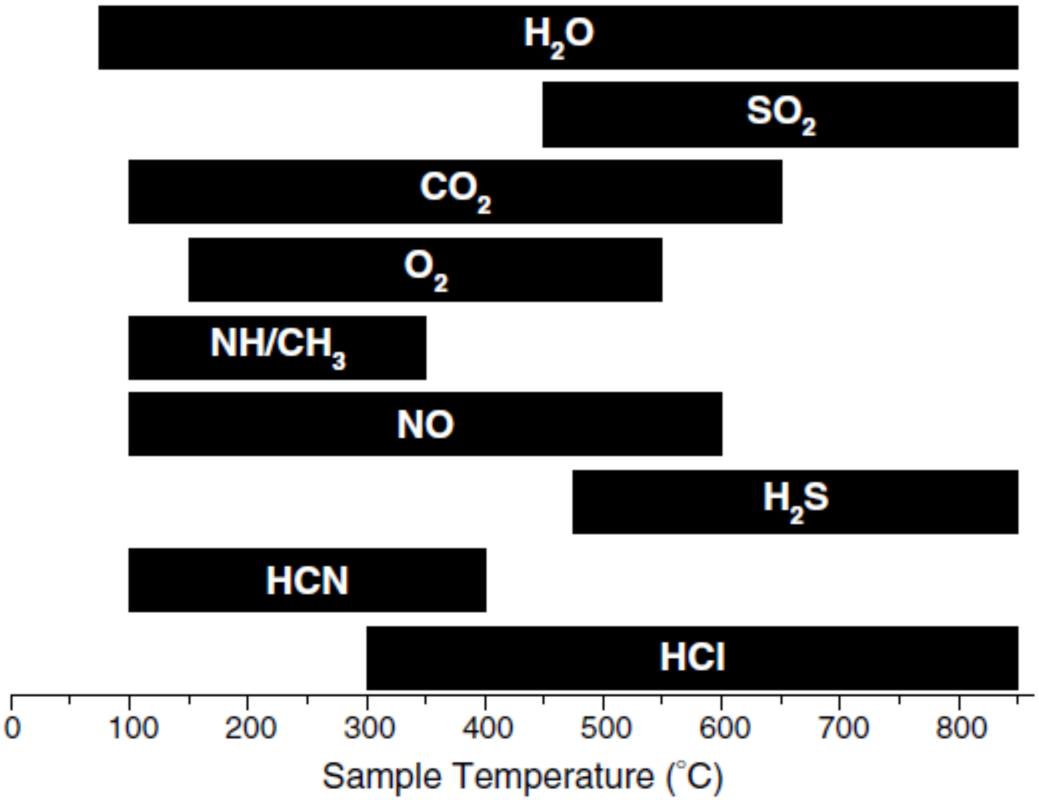


Figure 1. The primary gases evolved during SAM thermal analysis plotted versus the sample temperature range over which they were detected. Evolved gas analysis (EGA) products are arranged in order of decreasing molar abundance starting at the top.

"Abundances and implications of volatile-bearing species from evolved gas analysis of the Rocknest Aeolian deposit, Gale Crater, Mars", Archer, P D. Jr. et al J. Geophys. Res. Planets, 119, 237-254, 2014

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

APPROACH TO EXPLORING MICROWAVE ENERGY AS A METHOD OF CAPTURING WATER IN SITU

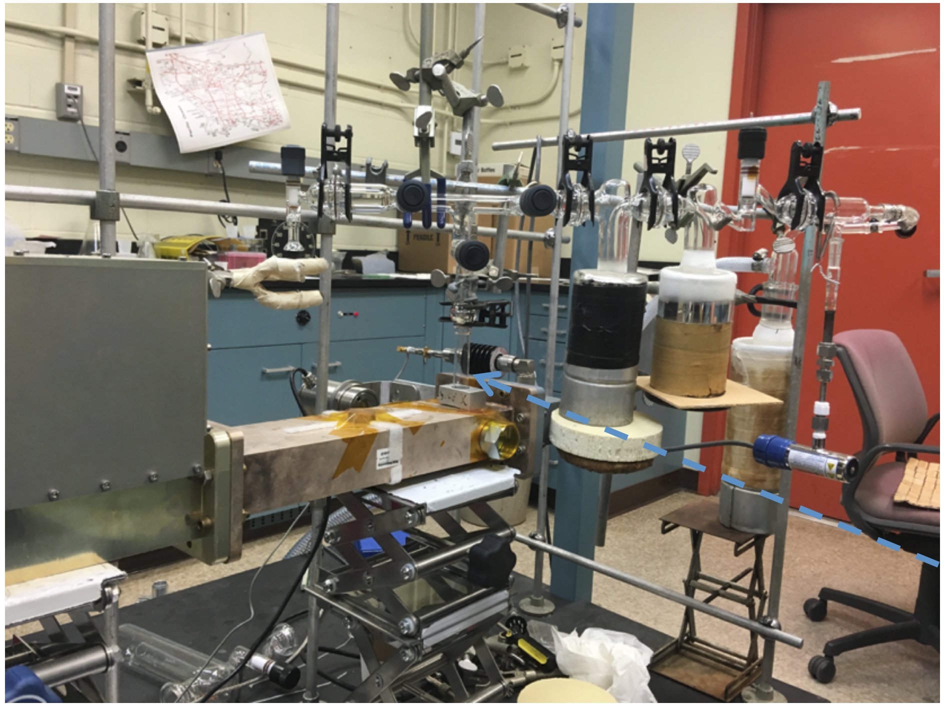
- **Determination of microwave effects specifically on water bound, either chemically or physically, critical to assessing applicability to ISRU**
  - Waters of hydration in salts, adsorbed surface water, ice, hydroxyl groups in clays, steam reaction with soil material all important factors to separate and ascertain for ultimately freeing water contained in soil
- **Poly-hydrated Sulfates and Clays were strong candidates according to M-WIP report**
  - Several types of sulfates and clays are considered primary sources of water
- **Phoenix findings suggested other candidate hydrates**
  - Perchlorates were identified as a highly probable constituent in Martian soil and easily acquired
- **Method for investigating microwave effects on water-containing material**
  - Initial work addresses use of microwave waveguide for baselining microwave operation conditions and test setup
  - Information crucial to designing advanced, practical microwave apparatus
    - Equipment design
    - Sample/regolith treatment processing approach

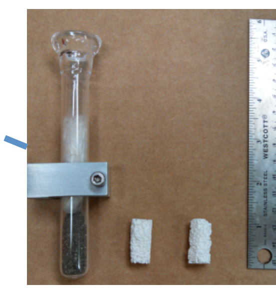
MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

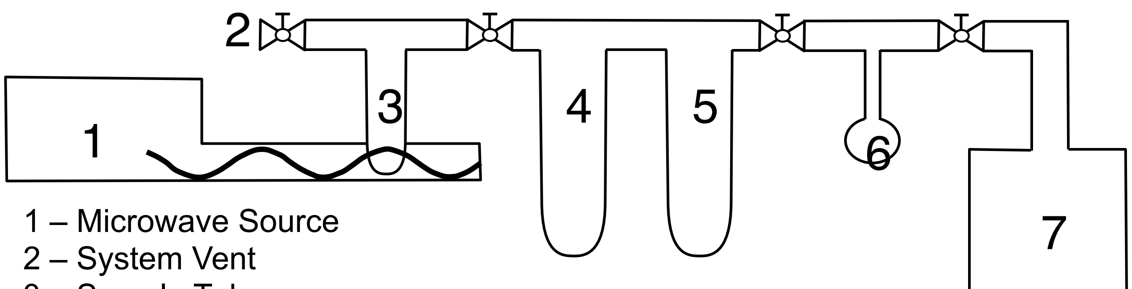
INITIAL APPROACH TO EXPLORING MICROWAVE ENERGY AS A METHOD OF CAPTURING WATER IN SITU

- **Microwave excitation system/power used**
  - WR340 waveguide cavity 34.67 cm long
  - 2.45 GHz microwave resonant mode frequency, using the transverse electric TE104 mode
  - Sample/holder positioned in the waveguide where the electric field is a maximum
  - 200 W Traveling Wave Tube amplifier (much larger than required for our current tests)
  - Labview software used to assist in maintaining resonant frequency during tests and recording microwave parameters
- **Sample types and sizes**
  - Samples contained in Quartz tube 1.6 cm OD (transparent to microwaves)
  - Samples (~ 1.3 cm ID, 2.54 cm long) contained known quantities of water-containing material
- **Collection strategy**
  - Capture water released during microwave excitation via selective LN_2 traps
  - Weigh water captured from sample treated during microwave excitation
  - Weigh material containing water before and after microwave treatment

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS







1 – Microwave Source
2 – System Vent
3 – Sample Tube
4 – Cold Trap 2
5 – Cold Trap 1
6 – Pressure Gauge
7 – Vacuum Pump

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

INITIAL APPROACH TO EXPLORING MICROWAVE ENERGY AS A METHOD OF IN SITU WATER DEVOLATILIZATION

- **Method for investigating microwave effects on water-containing material**
  - **Weigh sample to be treated by microwaves and place in quartz sample holder**
  - **Evacuate entire apparatus containing sample positioned in microwave cavity**
  - **Reach constant pressure before initiating microwave activation**
  - **Cool Trap #1 with LN_2 and initiate first level of microwave excitation for a given time**
  - **Cool Trap #2 with LN_2 and initiate second level of microwave excitation for a given time**
  - **At conclusion of microwave excitation, bring pressure in vacuum apparatus to atmospheric pressure with N_2 purge**
  - **Remove sample from apparatus and weigh each article to determine extent of water removal**

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

SEVERAL OBJECTIVES ASSOCIATED WITH INITIAL TESTS

1) Determine water recovery as a function of water-containing sample species
   - Correlate microwave power delivered to sample species release of water
2) Determine extent of water recovery (quantification)
   - Correlate quantity of water released with water captured
3) Determine water recovery as a function of microwave power
   - Correlate water released, water captured and power delivered to sample species
4) Determine water recovery as a function of microwave power and temperature of sample species
   - Correlate water released/captured with power delivered to, and temperature of, sample species
5) Determine water recovery as a function of regolith material
   - Examine effect of dielectric differences in sample material excited by microwaves

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

TEST OBJECTIVES

1) Determine water recovery as a function of water-containing sample species
   - Correlate microwave power delivered to sample species release of water

APPROACH

- Impregnated alumina foam support with hydrated salt sample species
  - Alumina foam does not absorb microwave
    - Permits understanding effect of microwave exciting water bound to sample species
  - Alumina foam does not inhibit release of water from sample species
  - Exact amount of water in sample species is easily determined for quantitation
  - Samples are easily aligned with microwave cavity to permit repeatability from test to test
  - Alumina foam can easily be impregnated with various hydrated salt materials

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

TEST OBJECTIVES

2) Determine extent of water recovery (quantitation)
   - Correlate quantity of water released with water captured

APPROACH

- System permits several means by which water released can be accounted
  - Vacuum established at beginning of each test can easily be determined and replicated
    - Vacuum conditions allows for easy transfer and capture of released water
  - Allows for an easy method by which water released under different conditions can be captured and related to test conditions
    - Dual traps allows for dual test conditions with same sample and operation
  - Both sample species and water collected can be weighed to account for water released due to microwave/temperature conditions of sample
    - Sample species is weighed before and after to determine water released
    - Total water contained can be determined by thermally driving all water from sample and weighing completely dehydrated sample species
  - System easily disassembled and reassembled for ease of test operations

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

TEST OBJECTIVES

3) **Determine water recovery as a function of microwave power**
   - Correlate water released, water captured and power delivered to sample species

APPROACH

- **Microwave power applied is an important function to relate to water release**
  - Microwave power can be set and maintained for prescribed time period
  - Apparatus is able to be tuned to maintain a chosen microwave power
  - Instrumentation allows experimenter to observe degree of microwaves absorbed versus reflected within waveguide during test
  - Changing power levels as a function of time period provide insight into relationship between microwave power absorbed and water released
  - Microwave power level can be changed with time for insight into rate and amount of water released

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

TEST OBJECTIVES

4) **Determine water recovery as a function of microwave power <u>and</u> temperature of sample species**
   - Correlate water released/captured with power delivered to, and temperature of, sample species

APPROACH

- **Both microwave power driven into waveguide and temperature of sample species are simultaneously monitored**
  - Microwave power level, and temperature of sample are both recorded
  - Correlation between microwave power, water released and energy absorbed by sample species are determined
  - Data from experiment permits a method by which microwave power required for water release from material and temperature of surrounding material can be correlated

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

TEST OBJECTIVES

5) **Determine water recovery as a function of regolith material**
   - **Examine effect of dielectric differences in sample material excited by microwave**

APPROACH

- **Monitor microwave power, time, temperature as a function of water released and type of material in sample species**
  - **Permits the determination of effect of water released and rate of temperature change to be correlated**
  - **Conducive to explaining differences between sample species dielectric properties, water release, power applied and temperature of sample material**
  - **Important to define relationship between microwave power required to devolatilize material, the extent of devolatilization and the time to achieve degrees of devolatilization**

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

Sample Name	Description	CT1-Cold Trap1 (g)	CT2-Cold Trap2 (g)	Σ Cold Trap (g)	% Recovered Based on Weight	Max. Temp °C
Epsomite Foam #1	2 W - 30min CT1 - 30min CT2	0.1379	0.0337	0.1716	77.69%	51
Epsomite Foam C	4 W - 15min CT1 - 15min CT2	0.0718	0.0456	0.1174	75.89%	51
Epsomite Foam D_a	10 W - 30min CT1 - 30min CT2	0.1212	0.0123	0.1335	77.39%	75
Perchlorate Foam B	2 W - 30min CT1 - 30min CT2	0.0064	0.0057	0.0121	4.34%	51
Perchlorate Foam D	5 W - 30min CT1 - 30min CT2	0.0076	0.0076	0.0152	7.38%	51
Perchlorate Foam A	10 W - 30min CT1 - 30min CT2	0.0665	0.0298	0.0963	48.74%	90
*Gypsum D	10 W - 30min CT1 - 30min CT2	0.0874	0.0720	0.1594	74.11%	122

*Gypsum was loosely placed in tube, not on alumina foam

Sample Name	Description	Cold Trap1 (g)	Cold Trap2 (g)	Σ Cold Trap (g)	% Recovered From CT	Max. Temp °C
**MMS+Gypsum B	3 W - 30 min CT1 - 30 min CT2	0.0831	0.0193	0.1024	94.91%	145

** MMS = Mojave Mars Simulant - surrogate Mars soil

[Power, temperature, frequency and pressure are continually monitored during run.]

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

INTERPRETATION OF EXPERIMENTAL WORK

- **Epsomite requires relatively minimal microwave power to release water**
  - First 30 minutes of 2 watts microwave released ~80% of recovered water
  - First 30 minutes of 10 watts microwave released ~90% of recovered water
  - Minimal temperature of hydrated material is required for high water content release
- **Magnesium perchlorate requires substantially more energy to release water**
  - Waters of hydration are more tightly bound than magnesium sulfate
- **Gypsum will require more time and/or power to release water**
- **Soil composition and water content variations may provide distinct differences in microwave impact on water release**
  - Dielectric potential of regolith must be considered in microwave energy absorption in water release of minerals
  - Clays and phyllosilicates must be examined for their contribution to overall soil water release

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

VALUE TO FUTURE ISRU PROJECTS

- Provides initial information to guide designs for ISRU applications in water recovery
  - Simplify means by which microwaves can be implemented in ISRU
- Provides insight into controls necessary for microwave applications to ISRU
  - Generate data necessary to achieve optimum energy utilization for control information guide
- Provides initial basis from which to determine efficiency advantages offered by microwaves in ISRU applications
  - Illustrates the need to better understand the relationship between material properties, water content, and microwave implementation for best application of microwaves
- Offers alternatives for microwave applications to early explorative adventures
  - Ease of introducing energy into regolith provides alternatives to conventional thermal methods

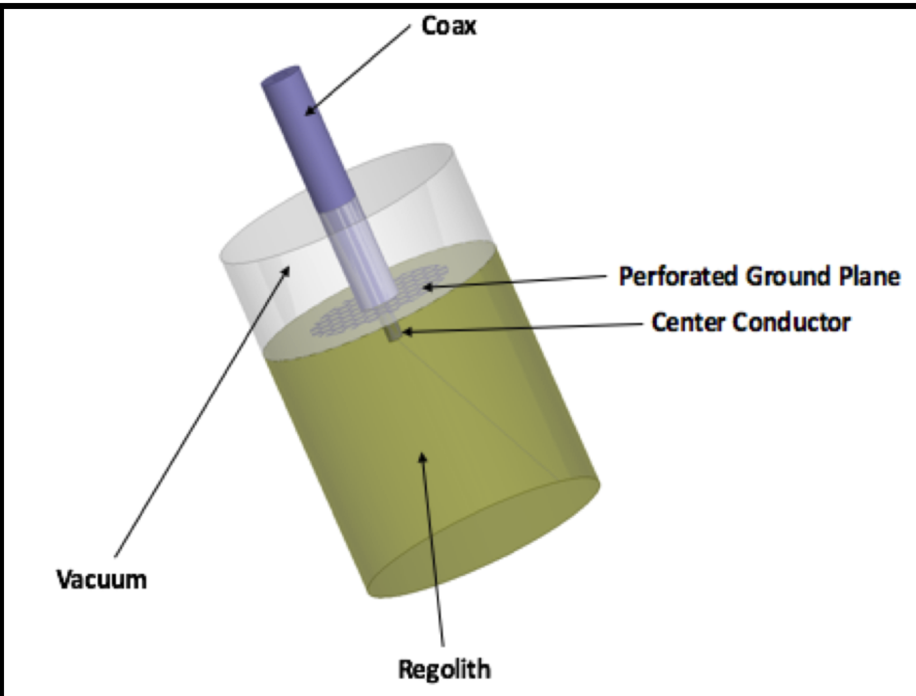
MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

PRELIMINARY CONSIDERATIONS TO APPLYING MICROWAVES TO ISRU

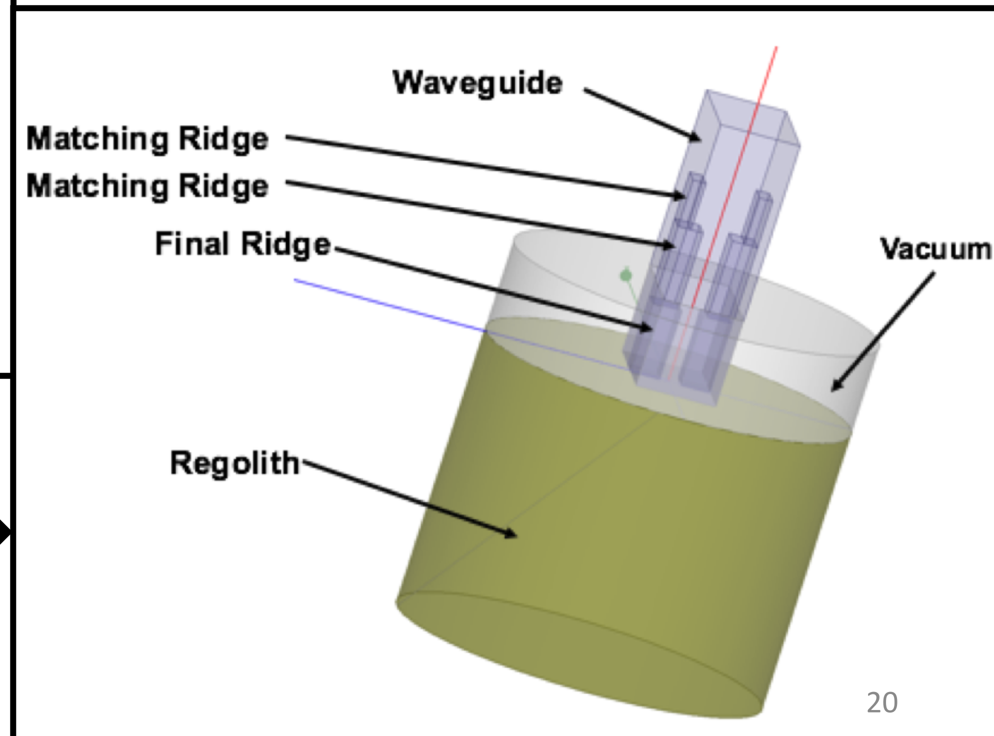
- **Perceived advantages of microwave application to regolith devolatilization**
  - Tune to primarily activate water held within regolith
  - Maintain regolith temperature at as low temperature as possible
  - Reduce the heat transfer limitation necessary for typical thermal treatment of regolith
  - More readily apply directly in situ to regolith, as opposed to large scale regolith transport
- **Various designs possible for applying microwaves to soil/regolith devolatilization**
  - Microwaves probe into regolith
  - Minimize temperature increase of regolith
    - Temperature will be a function of dielectric nature of regolith rather than heat transfer properties
- **Potential for rapid rate of water recovery**
  - Regolith can be systematically treated and returned to location with minimal transport
  - Extracted water can be collected in real-time and recovered onboard rover
- **Minimal introduction of heterogeneous volatiles into collected water**

MICROWAVE APPROACH TO EXTRACT WATER FROM HYDRATED MINERALS

TWO ATTRACTIVE METHODS FOR MICROWAVE USE



← Microwave Coaxial Cable Approach
Extending center conductor into regolith permits over 90% transmission of microwaves into regolith



Microwave Ridge Waveguide Approach →
Introduction of ridges allows over 90% transmission of microwaves into regolith without penetrating the regolith

NEXT STEPS IN EXPLORING USE OF MICROWAVE FOR ISRU WATER EXTRACTION

- **Investigate the power/rate of water release in samples**
  - Establish relationship between microwave initiation and maximum water extraction
- **Determine the actual energy required to extract water**
  - Correlate microwave energy, energy of water bound and environmental temperature relationships in water release
- **Examine effects of different regolith material composition effects on microwave power requirements**
  - Conduct controlled experiments with different surrogate material compositions
- **Explore different designs for employing microwaves in ISRU-applicable equipment**
  - Test conceptual designs to determine 'sphere of influence' and determine practical approaches to use of microwaves

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