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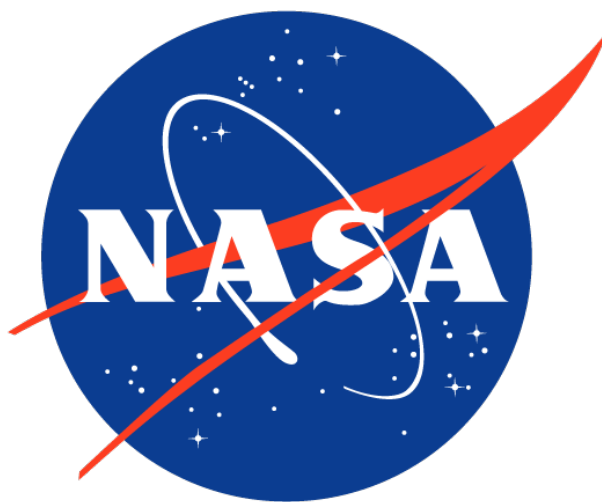**Instrument for Analysis of Organic Compounds on Other Planets**

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Instrument for Analysis of Organic Compounds on Other Planets

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The goal of this project is to develop the Instrument for Solvent Extraction and Analysis of Extraterrestrial Bodies using In Situ Resources (ISEE). Specifically, ISEE will extract and characterize organic compounds from regolith which is found on the surface of other planets or asteroids. The techniques this instrument will use are supercritical fluid extraction (SFE) and supercritical fluid chromatography (SFC). ISEE aligns with NASA's goal to expand the frontiers of knowledge, capability, and opportunities in space in addition to supporting NASA's aim to search for life elsewhere by characterizing organic compounds. The outcome of this project will be conceptual designs of 2 components of the ISEE instrument as well as the completion of proof-of-concept extraction experiments to demonstrate the capabilities of SFE. The first conceptual design is a pressure vessel to be used for the extraction of the organic compounds from the regolith. This includes a comparison of different materials, geometry's, and a proposition of how to insert the regolith into the vessel. The second conceptual design identifies commercially available fluid pumps based on the requirements needed to generate supercritical CO_2. The proof-of-concept extraction results show the percent mass lost during standard solvent extractions of regolith with organic compounds. This data will be compared to SFE results to demonstrate the capabilities of ISEE's approach.

Nomenclature

NASA	=	*National Aeronautics and Space Administration*
SFE	=	*Supercritical fluid extraction*
SFC	=	*Supercritical fluid chromatography*
ISEE	=	*Instrument for Solvent Extraction and Analysis of Extraterrestrial Bodies using In Situ Resources*
ISRU	=	*In Situ Resource Utilization*
KSC	=	*Kennedy Space Center*
SAM	=	*Sample Analysis at Mars*
TRL	=	*Technology readiness level*
ASME	=	*American Society of Mechanical Engineers*
JPL	=	*Jet Propulsion Laboratory*
CMS	=	*Ceralink Mars Simulant*
JSC	=	*Johnson Space Center*
Ace: Hex	=	*Acetone: Hexane*
FTIR	=	*Fourier Transform Infrared Spectroscopy*
PDL	=	*Prototype Development Lab*

I. Introduction

ISEE is an instrument that will characterize organic compounds within the regolith of planets, moons, or asteroids using in situ resources. Supercritical fluid extraction (SFE) and chromatography (SFC) coupled with mass spectrometry are the proposed methods used for the extraction, separation, and characterization of the organic compounds. The fluid that this instrument will use is carbon dioxide. In situ resource utilization (ISRU) is using the resources that the operating environment provides. Carbon dioxide utilization is an example of ISRU because CO_2 is

readily available during manned missions through exhalation or unmanned missions to Mars because its atmosphere is 95% CO_2 [1]. Different technologies for CO_2 capture and separation technologies for a Martian environment have been evaluated at Kennedy Space Center (KSC) [2].

NASA has previously developed technologies that characterize organic compounds from regolith [3], but ISEE is a competing approach because it uses in situ resources and has the potential to extract the full range of organic compounds (non-polar polyaromatic hydrocarbons to amino acids) as opposed to targeting one type at a time. The current unit analyzing organic material on Mars is the Sample Analysis at Mars (SAM) aboard the Curiosity Rover. The SAM unit is limited in its extraction capabilities because it only has limited supply of organic solvents for extractions of polar compounds. For non-polar compounds, SAM heats samples up to 1100 °C and collects them as vapors for analysis. ISEE aims to develop a technology that can process all organic material in one method without having a limited number of extractions while reducing mass. ISEE is currently at the first level of technology readiness (TRL 1) meaning that basic technology research questions must be answered before building an instrument. One research question addressed in this report is whether or not SFE is capable of extracting the full range of organics without using or requiring any consumables.

The different subsystems of the ISEE instruments include carbon dioxide capture from the environment, CO_2 pressurization and heating to a supercritical fluid, supercritical fluid extraction from regolith within a pressure vessel, supercritical fluid chromatography to separate the organics, characterization via mass spectrometry, and recycling the solvent. This report discusses conceptual designs of the pressure vessel, the fluid pump, and proof-of-concept extraction experiments to prove that SFE is capable of extracting the full range of organic compounds.

II. Instrument Design

A. Conceptual Design of Pressure Vessel

Preliminary research on conventional SFE pressure vessels was performed to gain an understanding of the functionality of pressure vessels. A pressure vessel is the component of the ISEE instrument where the extraction of the organic compounds from the regolith occurs. In this vessel, the carbon dioxide will flow in, be pressurized and heated to a supercritical fluid, dissolve the organic compounds, and exit the pressure vessel while carrying the organics for further analysis. Next, the necessities of the pressure vessel to fit ISEE's applications were identified. Specifically, the pressure vessel design must include a 10-mL vessel that is operable up to 276 psi and 100 °C to satisfy maximum SFE operating conditions. The vessel must also have inlet and outlet ports for CO_2. It must also have ports for the introduction and removal of regolith which should be confined within the vessel. Two different geometry's were considered in the study: a cylindrical and a spherical vessel, and the complete conceptual design can be seen in Figure 1.

1. Cylindrical Design

First, a comparison of different grades of aluminum, stainless steel, and titanium was done to determine the ideal material for the tubing of the pressure vessel. Three materials were selected that have high strength to weight ratios and have been used in aerospace applications.

Different metals have different strengths, so the required wall thickness of metal tubing at 276 psi and 100 °C was calculated for each metal in Table 1. The equation that was used for

Table 1: Minimum Wall Thickness for Metal Tubing

Material:	Outer Diameter (cm)					
	1.27	1.905	2.54	3.175	3.81	5.08
Titanium						
R50250	0.333	0.500	0.665	0.833	1.001	1.334
R50400	0.262	0.394	0.526	0.655	0.787	1.049
Stainless Steel						
S31600 (A269)	0.211	0.318	0.424	0.528	0.635	0.846
S32100	0.211	0.318	0.424	0.528	0.635	0.846
S30400 (A269)	0.211	0.318	0.424	0.528	0.635	0.846
S34700	0.211	0.318	0.424	0.528	0.635	0.846
Aluminum						
A95052	0.381	0.572	0.762	0.953	1.143	1.524
A95052-H34	0.297	0.447	0.594	0.744	0.894	1.191
A96061-T4	0.318	0.478	0.635	0.795	0.953	1.270
A96061-T6	0.264	0.396	0.528	0.660	0.795	1.059

the calculation of wall thickness follows NASA's technical standard for a straight pipe under internal pressure. This can be found in the American Society of Mechanical Engineers' (ASME) Code for Process Piping B31.3-2014

(paragraph 304.1.2). The calculations for required wall thickness and mass estimates were carried out with outer diameters of 1.27, 1.905, 2.54, 3.175, 3.81, and 5.08 cm (0.5, 0.75, 1.0, 1.25, 1.5, and 2.0 inches). A study performed by Langenfeld showed that the extraction rates do not significantly differ between extractions done in long and narrow vessels compared to short and wide vessels [4], so the geometry with the lowest mass is the best.

Mass is a driving factor in spaceflight applications, so the mass of the tubing required for a 10 mL vessel was calculated for each material as shown in Table 2. An outer diameter of 1.25" was chosen for Table 2; however, the excel program used for this task allows the user to manipulate the outer diameter. Table 2 shows that the lightest material is grade A96061-T6 of Aluminum for an outer diameter of 1.25 inches. This is also the lightest material for outer diameters of 1.27, 1.905, 2.54, 3.81, and 5.08 cm (0.5, 0.75, 1.0, 1.5, and 2.0 inches).

Current pressure vessels designed for SFE usually use a threaded end cap to seal cylindrical vessels. Placing the sample in the vessel involves unscrewing the end cap, inserting the sample inside a portable extraction thimble, screwing the cap back on, and manually removing the thimble after the extraction. A study preformed at NASA's Jet Propulsion Laboratory (JPL) suggests this method for supercritical fluid extractions from regolith. However, removable thimbles and unscrewing endcaps manually do not match ISEE's application.

Table 2: Mass of Metal Tubing Required for a 10 mL Pressure Vessel with a 3.175 cm Outer Diameter

Material	Wall Thickness Used (cm)	Density (g/cc)	Mass (kg)
Titanium (B861)			
R50250	0.833	4.51	0.155
R50400	0.655	4.51	0.086
Stainless Steel			
S31600 (A269)	0.528	8.00	0.100
S32100	0.528	9.01	0.113
S30400 (A269)	0.528	8.00	0.100
S34700	0.528	7.93	0.099
Aluminum (B210)			
A95052	0.953	2.68	0.141
A95052-H34	0.744	2.68	0.068
A96061-T4	0.795	2.70	0.081
A96061-T6	0.660	2.70	0.052

An alternative method for regolith insertion and removal from the pressure vessel was evaluated to increase the automated capabilities of ISEE and is shown in Figure 1. The new method is simpler and lends itself to automated operation. This idea includes having separate valves for regolith insertion and removal and permanent filters to confine the regolith once inside the vessel. A set up with 4 ports would work well with a spherical vessel which is considered next.

Lastly, fittings for the end caps of the pressure vessel were considered. The key factors for the selection of fittings are compatibility with tubing material, pressure ratings, and temperature ratings.

2. Spherical Design

The alternative method of having 4 fittings instead of unscrewing end caps fits best with a spherical geometry which is structurally durable. The conceptual design of the pressure vessel includes lateral inlet and outlet fittings for the CO_2 and top and bottom ball valve fittings for regolith to enter and exit through and can be seen in Figure 1. A spherical geometry was selected for the design of the pressure vessel. A spherical geometry would be ideal for this set up because the curved bottom would guide the regolith in dropping out of the valve after the extraction.

The valves suggested for the regolith ports are ball valves provided by Swagelok. Specifically, they are grade 316 stainless steel Multipurpose Ball Valves (AFS Series) with 3/4 inch end connections and are operable from -40 to 121 °C and up to 6000 psi. The connection size was chosen so that the valve opening is large enough for regolith to be funneled in, but small enough to mechanically attach to the spherical vessel. A CAD drawing of this design can be seen in Figure 1.

After comparing the size of the ball valves to a 10 mL spherical vessel, it was decided that a 10 mL vessel was too small to mechanically connect inlet and outlet fittings in addition to two ball valves. Therefore, a 25 mL vessel is the recommended volume for the conceptual design. The original 10 mL requirement was set as a minimum size for an extraction vessel to preform SFE capabilities, so a 25 mL vessel will satisfy the initial parameters. However, the 25 mL vessel will allow a design that does not require a vessel endcap to be removed to insert the regolith.

Permanent filters will be mechanically held in place inside the spherical vessel that cover the inlet and outlet fittings. These filters will allow the CO_2 to flow through the vessel but confine the regolith to the vessel. This design is advantageous because it allows for a built in non-disposable filter that increases the lifetime of ISEE and does not

wear on the end cap fittings. Selecting a filter will require considerations of the thermal limitations, stress limitations, and porosity. Suggested materials from Sterlitech Corp. include ceramic, silver, polycarbonate, and polyester filters.

The filters could either be held in place by the fittings or mechanically attached to the interior of the vessel in line with the fittings. Swagelok manufactures tube fittings (as shown in Figure 1) with varying outer diameters that meet operating pressures at ambient temperatures. However, the pressure ratings are not reported for ISEE's operating temperatures and are not designed to hold filters. Further investigation of mounting the filters and selecting a tube outer diameter is required. The vessel material recommended for the conceptual design is S31600 stainless steel in order to be compatible with the fittings.

Table 2 shows that the overall lightest material is Aluminum A96061-T6. However, for a 10 mL cylindrical tubing, every tube weighs less than 0.20 kg. As mentioned before, the SAM aboard the Curiosity rover at Mars is the technology ISEE is competing with and weighs 40 kg [3]. If the mass of an aluminum vessel was significantly less than a stainless steel vessel, then Aluminum would have been used in the conceptual design. However, the mass of the vessel material is not significant enough to be a driving factor in the design of the pressure vessel.

Figure 1: CAD Drawing of a 25 mL Spherical Pressure Vessel with Ball Valves

B. Conceptual Design of Pump

In the conceptual design of ISEE's pump, the key factors of the pump were identified and commercially available pumps were evaluated. A pump is needed in two different steps of ISEE: the extraction and the chromatographic system. The feasibility of using one pump that meets all of ISEE's needs was evaluated by considering the fluid requirements for the extraction and SFC. The mass of the pump should be below 10 kg in order to compete with the SAM technology. A pump for the extraction must deliver CO_2 at 138-276 bar (2000-4000 psi) with a flow rate near 2 mL/min. The range of pressure are essential because different temperature and pressure conditions target specific organics [5]. The pump for SFC must pressurize CO_2 to 2000 psi and must deliver the fluid at a constant flow rate of 5 mL/min due to the importance of retention time in a chromatographic system. The pump is not involved in heating the fluid. Standard SFE systems operate by surrounding the vessels that require supercritical conditions with a heater to maintain supercritical temperatures [6].

During the conceptual design of the pump, the use of a modifier within the system was also considered. A modifier is a co-solvent that is used in SFE and SFC applications to assist in the extraction and separation of polar compounds. A modifier affects the conceptual design of the pump because a second pump would need to be used for the compression of a modifier. Then once the CO_2 and the modifier are at the same temperature and pressure, they are combined to create a homogenous supercritical fluid.

Three different pumps are commonly used in supercritical fluid systems: pneumatic amplifier pumps, syringe pumps, and reciprocating pumps [4]. Pneumatic amplifier pumps are not practical for ISEE's application because they require the flow of pressurized gas (usually air) to a piston which drives the fluid and are not accurate enough for SFC [7]. Syringe pumps operate by filling a reservoir with a fluid and controlling the delivery through a moveable piston. Syringe pumps are ideal for ISEE's application because they allow for a wide range of flow rates. However, once the reservoir volume is swept, it needs to refill resulting in a disrupted flow rate unless a 2-syringe pump is used. Lastly, reciprocating pumps used for SFC and SFE can deliver a consistent flow, but it has been noted that they require the cooling of a pump head which is not ideal for ISEE's application [7].

Six different commercially available syringe pumps are summarized in Table 3 on the next page.
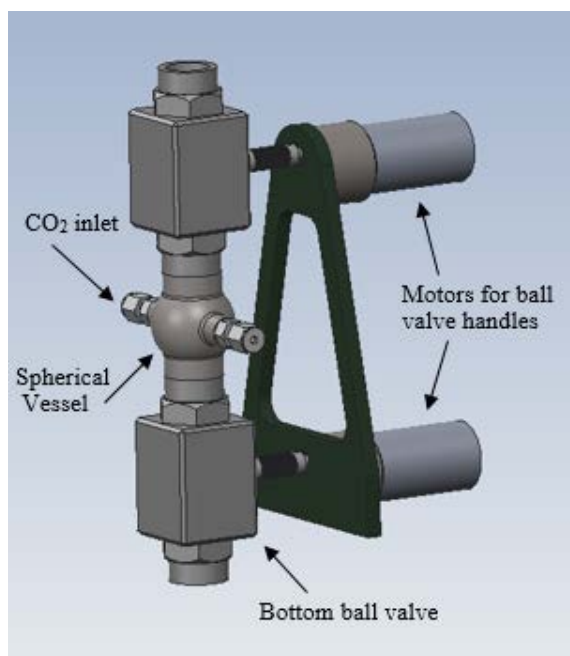
Table 3: Commercially Available Syringe Pumps

Company	Model	Dimensions (cm)	Mass (kg)	Max Pressure (bar)
Supercriticalfluids.com	SFT Series I Plus	15 x 20 x 33	8.6	345
Cetoni	5 mL Nemesys High Pressure	30 x 10 x 15	4.5	228
Cole-Parmer	115 VAC High Pressure	15 x 25 x 28	7.3	241
Supercriticalfluids.com	SFT-10	15 x 28 x 53	17	689
Isco	100DX	104 x 28 x 46	36	689
New Era Pump Systems	NE-8000	28 x 15 x 15	3.5	69

The pumps supplied by Cetoni, Cole-Parmer, the SF-10 model provided by supercriticalfluids.com, Isco, and New Era Pump Systems were eliminated because they did not meet the initial pressure or mass requirements. Only the SFT Series I Plus pump supplied by supercriticalfluids.com is capable of meeting the initial pressure and mass requirements. However, this pump is only capable of compressing one fluid at a time. A modifier will most likely need to be used for the extraction and separation of the polar organic compounds such as amino acids. Therefore, even the SFT Series I Plus model is impractical for ISEE's application because two SFT Series I Plus pumps would be needed which would exceed the mass requirement for the pump.

No commercially available options match ISEE's pressure, mass, and application needs. Manufacturing a pump that meets ISEE's application is possible at KSC. Therefore the fluid pump could be designed at the Prototype Development Lab (PDL) at KSC.

The complete conceptual design of the fluid pump includes a syringe pump with three syringes. Two syringes would be used pump CO_2 and the third would be used for the modifier. Two syringes are needed for CO_2 because a constant flow rate is required which cannot be achieved with only one syringe. However modifiers typically are added to near 5% concentration, so only one syringe would be needed for the compression of the modifier. Two back pressure regulators will be placed in line with the fluid flow to ensure that pressures are maintained. One regulator will be after the extraction vessel and the other is needed after the chromatographic columns.

III. Proof-of-concept Extraction Experiments
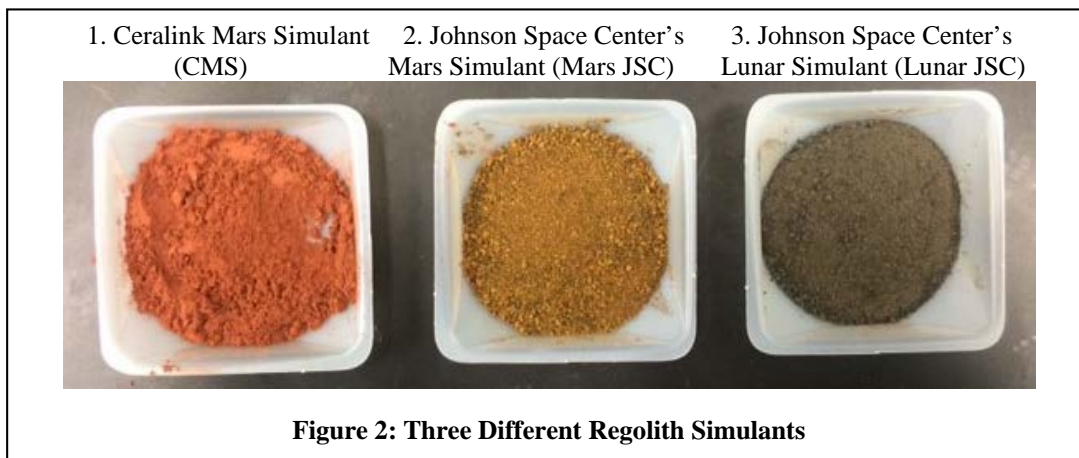
A. Extractions of Regolith Simulants

Three simulants were chosen for extraction tests. Prior to running SFE, the three regolith simulants were characterized for moisture content and organic material using traditional methods. Moisture content was measured by drying simulants in a 105 °C oven until a stable mass was reached to determine the percent moisture content. The results are shown below in Table 4.

Table 4 shows that the Mars JSC simulant has the highest moisture content. Because the JSC Mars moisture content is almost 10% by mass, we would expect some of this content to be extracted out during Soxhlet extractions. Soxhlet extractions refer to using an automated machine that preforms liquid extractions on a sample that is held in an extraction thimble. The CMS and Lunar JSC simulants have much smaller moisture contents; therefore, we would expect less mass to be extracted from these simulants.

Table 4: Moisture Content of Three Regolith Simulants

Simulant	Moisture Content (%)
CMS	1.51
Mars JSC	9.51
Lunar JSC	0.08

Soxhlet extractions were performed to determine the amount of organic material in each simulant. The simulants used are shown in Figure 2.

1. Ceralink Mars Simulant (CMS)   2. Johnson Space Center's Mars Simulant (Mars JSC)   3. Johnson Space Center's Lunar Simulant (Lunar JSC)

Figure 2: Three Different Regolith Simulants

Three extractions of each simulant were run for a total of nine extractions. Whatman cellulose, 25 x 100 mm extraction thimbles held 10-15 g of regolith simulant during the extractions. The extractions were performed using 75 mL of a 1:1 by volume solution of Acetone: Hexane (Ace: Hex) as the solvent. This solution was chosen because it is used in standard EPA methods. The extractions ran for two hours with heating parameters recommended for the selected solvents by the Buchi Soxhlet extractor manual.
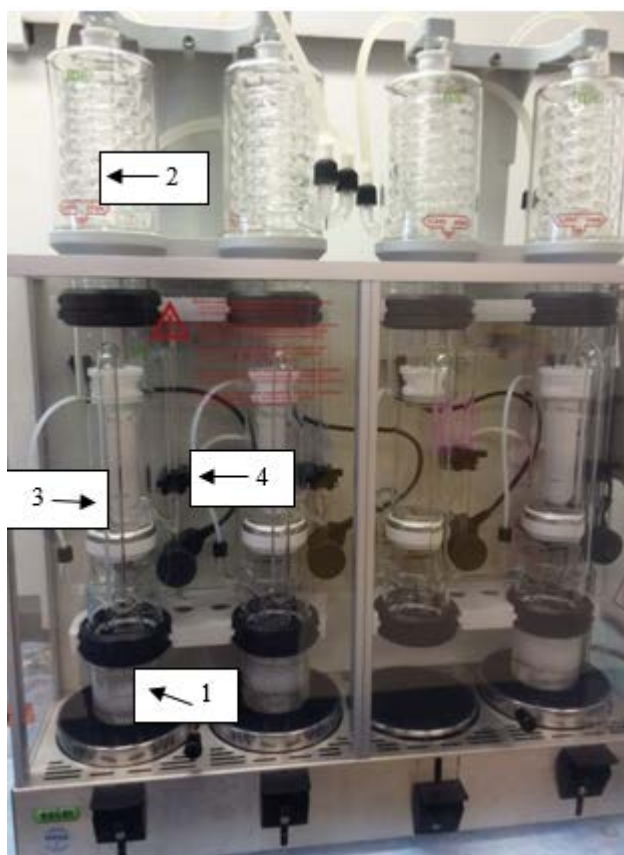
There are four main steps duirng a Soxhlet extraction as shown in Figure 3. During step 1, the solvent is heated and evaporates through the glass tubing up to the condenser coils. Second, the solvent is exposed to a cold fluid loop and condenses into the chamber with the extraction thimble. Next, the condensed solvent fills up around the extraction thimble and dissolves the organics by filtering in and out of the thimble. Lastly, once the optical sensor is activated, a valve is automatically opened and the solvent is recycled along with the organics to the solvent cup. A two hour extraction will repeat the four steps about 25 times.

The mass of the regolith simulants were taken before the extraction and after drying the simulants in a fume hood overnight. The mass of the thimbles were checked a few days after to ensure stable thimble masses. The masses before and after the extractions were compared to determine the overall percent mass lost from standard extractions of uncontaminated simulants. The results are shown in Table 5 on the next page.

Figure 3: An Ongoing Soxhlet Extraction with ¾ Chambers Running

**Table 5: Soxhlet Extraction Data of
Three Regolith Simulants**

Simulant	Average Change in Mass (%)
CMS	0.44 ± 0.085
Mars JSC	-2.4 ± 0.12
Lunar JSC	0.12 ± 0.044

Table 5 shows that the Mars JSC simulant lost an average of 2.4% by mass during the Soxhlet extractions, but the other two simulants gained less than 1% of mass. The increase in mass in the CMS and the Lunar JSC simulants could be due to solvent that got absorbed by the regolith. It is plausible that small amounts of organic solvent did not evaporate in the fume hood overnight due to confinement in the extraction thimble and ambient temperatures.

B. Extractions of Spiked Regolith Simulants

Soxhlet extractions were conducted on a simulant spiked with organic material that would be expected to be seen in extraterrestrial samples. This will serve as a comparison to the SFE results to determine whether or not SFE is capable of extracting the full range of organic.

Three organic compounds that represent the full range of organics that ISEE hopes to characterize were selected as test compounds for extractions. These compounds were naphthalene (polyaromatic hydrocarbon), stearic acid (long-chain carboxylic acid), and L-tryptophan (amino acid). The lunar simulant was selected for the proof-of-concept extraction experiments of a spiked regolith simulant. It was chosen because it has the lowest moisture content as shown in Table 4, and it had the least percent change in mass during uncontaminated Soxhlet extractions shown in Table 5. The low moisture and organic content will simplify the comparison with SFE.

Organic compounds were added to the simulant according to the following procedure: the solid organics were dissolved in an organic solvent, the solvent was added to the regolith, and then the solvent was evaporated using a Rotovapor as seen in Figure 6. It was essential to dissolve the contaminants in an organic solvent before adding them to the regolith to ensure a homogenous mixture. Naphthalene and stearic acid were dissolved in acetone, and L-tryptophan was dissolved in methanol. The contaminants were added to each have a concentration of 0.10 g of each contaminant per extraction thimble which holds about 10 grams of regolith. Once a homogenous mixture of regolith simulants spiked with organic compounds was made, an extraction of a contaminated lunar simulant was performed with a 1:1 mixture of Ace: Hex.

The extraction efficiency of the contaminated lunar simulant with Ace: Hex is shown in Table 6. This efficiency represents the amount of contaminants extracted compared to the amount of contaminants present in the regolith. It is clear that the Soxhlet extraction using Ace: Hex did not successfully extract all of the organics. It is expected that the stearic acid and naphthalene were successfully extracted, but the tryptophan was not due to the solvent not being polar enough to dissolve L-tryptophan. Therefore, a second extraction was performed on the same extraction thimbles using Methanol. It is expected that the methanol would successfully extract the remaining tryptophan in the thimbles. As shown in Table 6, the extraction efficiency of the contaminants increases after the second extraction about 20%.

**Table 6: Extraction Efficiencies for
Two Lunar Extractions**

Fraction of Contaminants Extracted after Ace: Hex Extraction (%)	Fraction of Contaminants Extracted after Methanol Extraction (%)
54.7 ± 8.61	84.3 ± 7.15

A Fourier transform infrared spectroscopy (FTIR) analysis was performed in order to determine which compounds were being extracted by each solvent. Figure 4 compares the transmittance spectrum of the extract recovered after a 2 hour Soxhlet extraction using a 1:1 by volume solution of Ace: Hex to a standard stearic acid sample. This spectrum confirms that the collected extract after the first extraction is primarily stearic acid. Naphthalene should be present in the top spectrum; however, the FTIR was taken a few days after the extraction and it is probable that the naphthalene evaporated away due to its high vapor pressure.
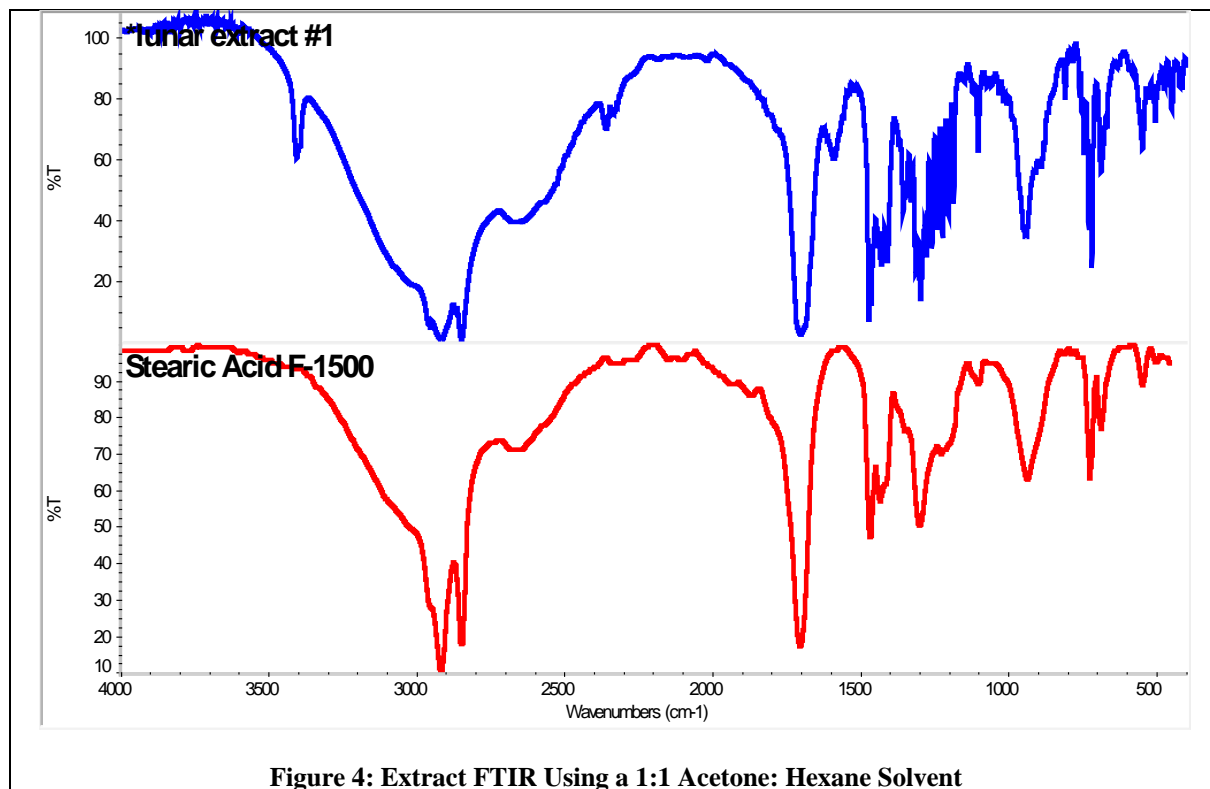
Figure 4: Extract FTIR Using a 1:1 Acetone: Hexane Solvent

Figure 5 compares the IR spectrum of the collected extract from the methanol extraction to a standard L-tryptophan spectrum. It is clear that L-tryptophan is the primary compound processed in the methanol extract.
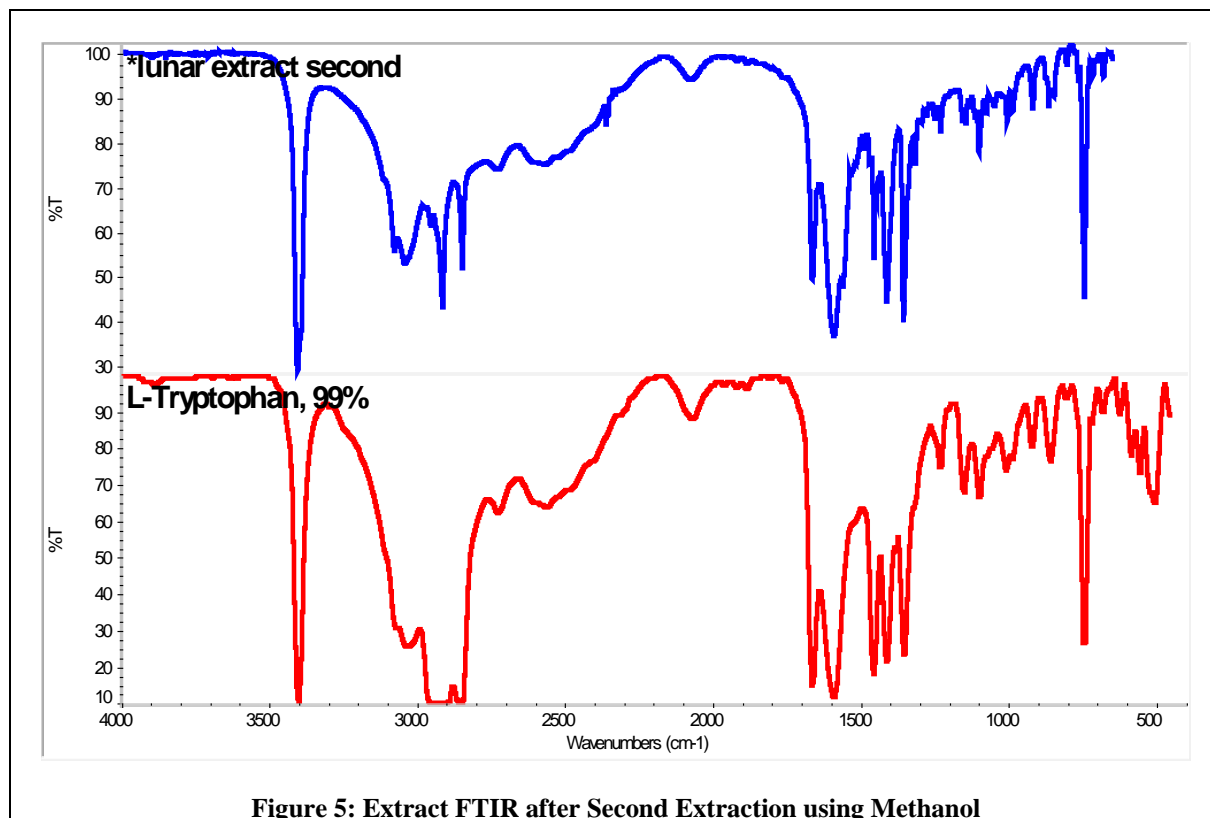

Figure 5: Extract FTIR after Second Extraction using Methanol

As shown in Table 6, the percent of contaminants extracted compared to the amount of contaminants within the samples after the 2 extractions is still only 84.3% as an average of three trials. This lower than 100% recovery could be a result of the naphthalene evaporating at some point during the process. It could also be a result of an uneven distribution of contaminants or extraction limitations of Soxhlet extractions in general because the samples are never stirred during the extraction.

Figure 6: Acetone Evaporating from a Spiked Lunar Simulant Using a Rotavapor

Since the extraction efficiency increased by 20% after the methanol extraction, it was decided that Soxhlet extractions of contaminated regolith simulants would be carried out by first running a 2 hour extraction with Ace: Hex followed by a 2 hour extraction with methanol. The contaminants were added to each have a concentration of 0.10 g of each contaminant per 10 grams of regolith for the rest of the extractions. To further expand the range of organics used for contamination, polystyrene, a polymer with an aromatic ring, was also used as a contaminant for the proof-of-concept Soxhlet extraction experiments of the contaminated lunar simulant.

The batch of contaminated lunar simulant was spiked to have a 4% by mass contamination. This concentration allows 0.1 g of contaminant per extraction thimble to ensure that detection limits are met. The contaminants added to this batch of lunar regolith are L-tryptophan, polystyrene, stearic acid, and naphthalene. The L-tryptophan was added first by dissolving it in methanol and evaporating the methanol. The rest of contaminants were added by dissolving in acetone which was then evaporated as shown in Figure 6. A 300 gram batch of contaminated lunar simulant was made for the Soxhlet extractions and will be used in future SFE runs. Two trials of 3 extraction thimbles were performed for a total of 6 Soxhlet extractions of contaminated lunar regolith simulant. Extractions were performed with a 1:1 solution of Ace: Hex and then methanol. This method ensures that the full range of organics are extracted and the data is complete to compare SFE results to. The results are summarized in Table 7 below.

Table 7: Soxhlet Extraction Data of Contaminated Lunar Simulant

Trial	Change in sample mass during extraction / Mass of contaminants in sample (%)	Grams recovered from extraction / Change in sample mass during extraction (%)	Grams recovered from extraction / Grams regolith (%)
1	76.44	97.4	2.977
2	77.39	97.4	3.017
3	75.98	96.0	2.919
4	78.03	95.8	2.991
5	77.68	99.4	3.088
6	78.29	95.3	2.985
Avg:	**77.3 ± 0.911**	**96.9± 1.48**	**3.00 ± 0.0553**

Table 7 shows that an average of 77.3% of contaminants were extracted during the Soxhlet extractions. This yield could be a result of contaminants evaporating during the process. If the contaminates evaporated during this stage, then the concentration of contaminants would be less than what was assumed for the calculations in Table 7. Extracting less than 100% of contaminants during the Soxhlet extractions could also be a result of limitations of the

Soxhlet method because there is no stirring mechanism. Table 7 also reports that an average of 96.9% of extracts were recovered. This value validates the recovery methods and confirms that solid organic compounds were extracted. Lastly, Table 7 reports the fraction of grams recovered from the each sample mass and was calculated to have an average of 3.00%. This value is valuable because recovering and characterizing extracts is the ultimate goal of ISEE. The fraction of grams recovered from a sample of regolith will serve as comparing data to SFE recovery results.

IV. Conclusions

A conceptual design of a pressure vessel to be used for the extraction of organic compounds from regolith was performed in this report. This resulted in a design of a 25 mL stainless steel, spherical vessel with 2 lateral fittings for CO_2 flow and two vertical ball valves for the entrance and exit of the regolith. A conceptual design of a CO_2 fluid pump to be used for the compression of carbon dioxide for supercritical fluid extraction and chromatography was also performed. Six commercially available fluid pumps were evaluated that met ISEE's application, and no pumps were found to meet the pressure, size, and mass requirements. Therefore it was decided that if the TRL of ISEE increases to 5 and a flight-like model of ISEE is built, then a pump that meets ISEE's requirements would be designed at KSC. A potential set up for this pump is a syringe pump with three syringes. Two syringes would compress CO_2 and the third would be used for a co-solvent.

Proof-of-concept extraction experiments were performed to generate data to compare SFE results to. These extractions showed that Soxhlet extractions successfully extracted an average of 77.3% of the organic contaminants from the lunar regolith simulant with an average of 96.9% recovery of those contaminants. The recovery of the organics is especially important to compare to the SFE results because recovery and characterization of organic compounds is the goal of ISEE. Specifically, the data that will be compared to SFE results is the fraction of grams recovered of the contaminated regolith sample. Six Soxhlet extractions showed that an average of 3.00% by mass was recovered from the regolith samples.

So far, a conceptual designs of the pressure vessel, the pump, and Soxhlet extractions to generate data to compare SFE results to have been completed. Moving forward, the conceptual designs will provide a framework for the design of the instrument. However, before completing a holistic design of the ISEE instrument, the SFE and SFC approach has to be proven to be effective in extracting and separating organic compounds from regolith. This will be done in summer of 2016. This report resulted in standard extraction data that will serve as a comparison to the SFE results. If the SFE extraction efficiencies match the Soxhlet extraction results, then ISEE's approach can be supported. If this is true, ISEE will increase to TRL 2 and the next fundamental research question can begin to be answered. For example, the purity of CO_2 needed for SFE and SFC will need to be determined. This research question is essential to answer because CO_2 capture from the environment is the method of obtaining the solvent.

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