

# Miniature Cryogenic valves for a Titan Lake Sampling System

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**Abstract** – The Cassini mission has revealed Titan to be one of the most Earthlike worlds in the Solar System complete with many of the same surface features including lakes, river channels, basins, and dunes. But unlike Earth, the materials and fluids on Titan are composed of cryogenic organic compounds with lakes of liquid methane and ethane. One of the potential mission concepts to explore Titan is to land a floating platform on one of the Titan Lakes and determine the local lake chemistry. In order to accomplish this within the expected mass volume and power budgets there is a need to pursue the development for a low power lightweight cryogenic valves which can be used along with vacuum lines to sample lake liquid and to distribute to various instruments aboard the Lander. To meet this need we have initiated the development of low power cryogenic valves and actuators based on a single crystal piezoelectric flexensional stacks produced by TRS ceramics Inc. Since the origin of such high electromechanical properties of Relaxor-PT single crystals is due to the polarization rotation effect, (i.e., intrinsic contributions), the strain per volt decrease at cryogenic temperatures is much lower than in standard Lead Zirconate Titanate (PZT) ceramics. This makes them promising candidates for cryogenic actuators with regards to the stroke for a given voltage. This paper will present our Titan Lake Sampling and Sample Handling system design and the development of small cryogenic piezoelectric valves developed to meet the system specifications.

**Keywords:** Actuators, Piezoelectric Devices, Cryogenic valves, Titan, Liquid sampling

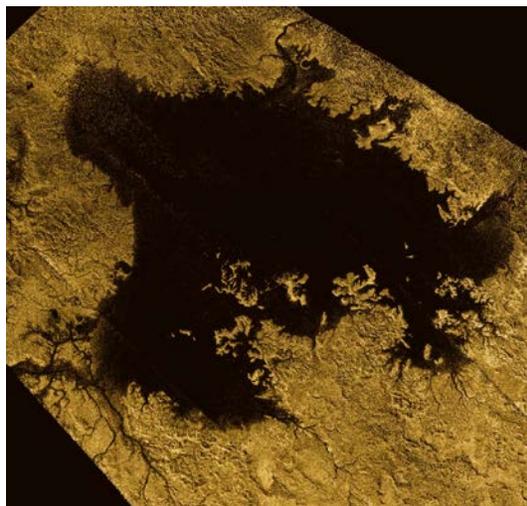
## I. INTRODUCTION

Since its arrival in Saturn orbit, the Cassini mission has found Titan to be one of the most Earthlike worlds in the Solar System (see Figure 1), The surface features have many of the same structures found on earth including lakes, river channels, playas, evaporite basins, and dunes<sup>1</sup>. In comparing to Earth however the materials and fluids which compose Titan features are organic compounds<sup>2,3</sup>. The analysis of Cassini VIMS spectral data<sup>2</sup>, found little evidence for exposed water ice on Titan's surface; instead they found that the reflectance spectrum of Titan best matches a mixture of organic compounds. The organic surface materials are derived from complex photochemical reactions that occur high in Titan's atmosphere<sup>4,5</sup>. The reaction products drift down to the surface to create deposits that are subjected to rains composed of fluids such as methane, nitrogen, and ethane<sup>6,7</sup>. At Titan surface pressures and temperatures a mixture of liquid methane and photochemically-produced ethane is the most likely liquid, with a significant amount of dissolved nitrogen. This mixture can serve as a solvent for the complex mixture of photochemical products formed in the upper atmosphere. Observations by Cassini provided evidence of a dynamic meteorological hydrocarbon cycle that is analogous to the water-based hydrological cycle on Earth.

Like Earth, the surface of Titan presents a wide variety of chemical environments, the result of a thick atmosphere and a multitude of possible geological processes active on Titan's surface. These various chemical environments provide a variety of settings for chemical partitioning and reactivity on the surface and the thick atmosphere.

Titan's lakes are a primary target for future Titan exploration, and serve as a nexus for the complex chemical interchanges between the surface and the atmosphere. Ligea Mare (Figure 1) was the landing target for the proposed TiME Titan lake lander. The lakes are a repository for the products of atmospheric chemistry, interconnecting Titan's geology, atmosphere, and astrobiological potential. Detailed chemical analysis is thus a goal of any mission to the lakes. Standard organic analysis

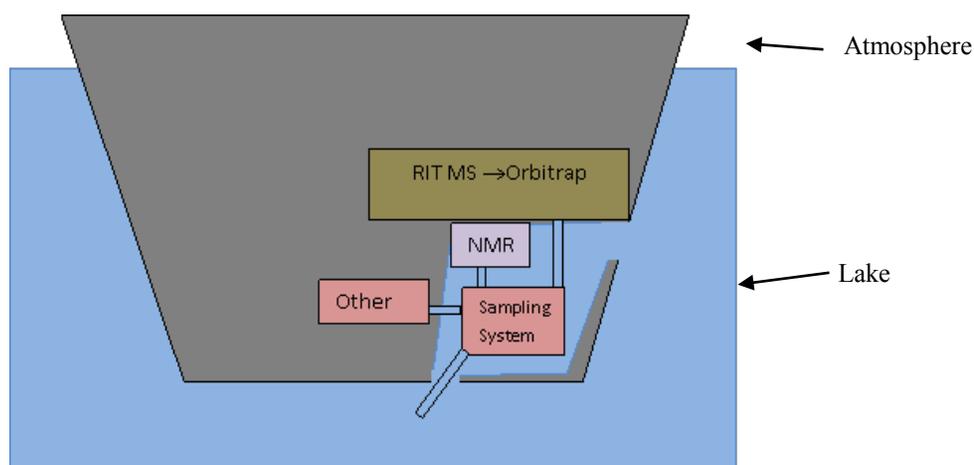
techniques, such as gas chromatography and mass spectrometry (used by the petroleum industry in the analysis of liquid natural gas), require acquisition and transfer of a sample to the analytical instrument.



**Figure 1:** Colorized Cassini RADAR image of Ligea Mare, a lake on Titan that is larger than Lake Superior on Earth. Planetary Photojournal PIA17031 (NASA/JPL-Caltech/ASI/Cornell).

The sampling system should have the ability to accommodate potential solid sample from beneath the spacecraft however this paper will focus on the liquid sampling and distribution system. To the best of our knowledge, no cryogenic liquid sampling and distribution system suitable for Titan’s surface exploration has been developed that addresses all of the features of the system we describe in this paper. The ability to sample filtered and unfiltered lake liquids without producing a phase change is critical to the determination of the accurate physical chemistry of Titans Lakes.

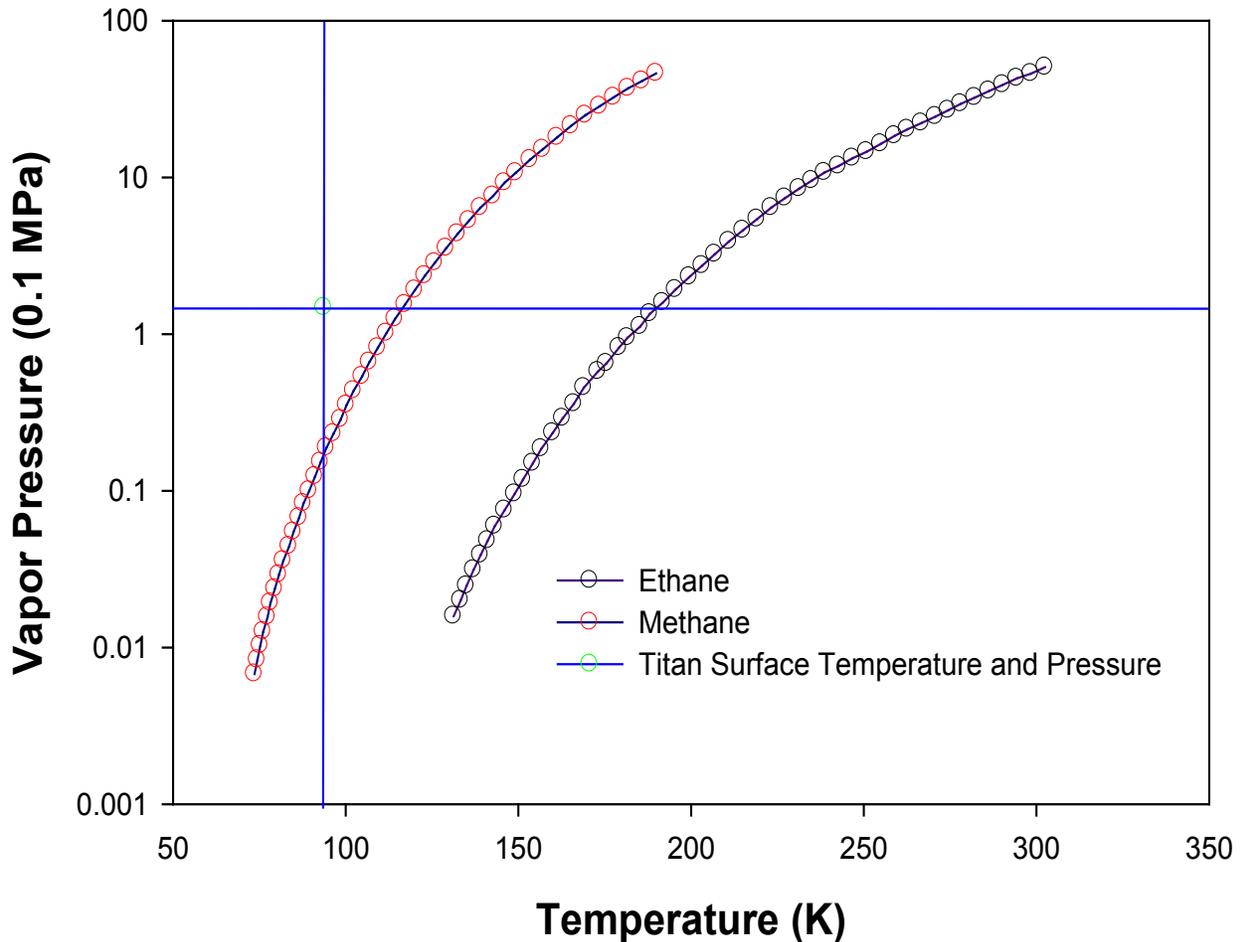
The proposed sampler and sample distribution system will be applicable to testing both in the lakes and on land, where using a common distribution design will allow for minimizing the mass and complexity of the system. The sampler will allow the determination of the physical chemistry of the liquid hydrocarbon reservoirs and the shoreline. Further, this system allows for the interrogation of samples by multiple instruments to enhance our understanding of the lakes and their role in the global hydrocarbon cycle of Titan. A schematic showing a cross section of the sampler concept is shown in Figure 2 along with potential instruments.



**Figure 2:** A Schematic of the floating spacecraft, sampling system and potential instrument suite.

This distribution system for the in-situ Titan lake sampler incorporates low power low mass piezoelectric actuators for cryogenic valving. The technology will not be limited in its capability to Titan since it will be applicable to characterization of the subsurface of other planetary icy bodies in the solar system [eg. Tethys, Enceladus, Europa] where liquid may exist or be generated from frozen solids. In the case of a solid ice/hydrocarbon surface such as may be found on a comet or other icy body the developed system could potentially act as a sample handling and sample distribution system between the sampler and the in-situ instruments or sample return storage vessels.

The Lakes of Titan are thought to be primarily composed of methane and ethane which are in thermal equilibrium with the atmosphere and surface. The vapor pressures of methane and ethane are important for the effective design of an ambient phase sampler and are shown in Figure 3. The surface pressure and temperature of Titan is 1.47 atmospheres and 93.7 Kelvin. The boiling temperature (when the vapor pressure equals the ambient pressure) of the methane and ethane would occur at 110 K and 190 K respectively. This means that thermal control of the sampling system is critical for the controlled sampling and the heating each of the liquids must be small or be accounted for by the heat capacity ( $\approx 2$  kJ/kgK at 90K) of the methane and thermal and the heat conductivity in the fluid away from the spacecraft.



**Figure 3:** The vapor pressure of Methane and Ethane and the surface pressure and temperature of Titan.

## II. BENCH TOP DESIGN

An example of a bench-top sampling system designed and tested at JPL that has the required functionality is shown in Figure 4 along with a schematic diagram of the valving and the activation sequence chart for each sampling state. The system was built to demonstrate the required functionality of a Titan Sampling system for sample distribution to three separate instruments. Not shown in the figures is the vacuum manifold that supplies the vacuum to the inlet ports of each instrument. The solid sampler which is not shown has been designed based on a narrow pneumatic penetrator that samples the solid and returns the sample to the spacecraft where it is melted under controlled conditions. The sample outlet of the solid sampler is connected to the inlet of the liquid sampler allowing for a single solid sample to be delivered by melting to the liquid sampler and sample handling system design. A variety of thermal problems need to be addressed when sampling from a cryogenic lake. The first is the heat flow from the spacecraft to the local fluid. The outer surface of the spacecraft is in contact with the methane and ethane of the lake. Any significant heating from the spacecraft will produce boiling. As such pulling the lake liquid into the sample handling system must be done under thermal control to prevent flash evaporation of the sample. Heat flow from the outer surface and the sampling chamber surface must be limited to ensure that the temperature at the spacecraft surface remains below the vapor pressure of methane. In addition the power dissipated by the valving sample distribution system must be conducted away from the sample.

The system has four different sampling states for the solid and liquid samples depending on which valves are actuated. The system can for the given instruments discussed:

- Draw unfiltered fluid to an optional instrument.
- Draw filtered fluid to the NMR.
- Draw filtered fluid to the Rectilinear Ion Trap (RIT)/Orbitrap inlet.
- Draw the filtrate solution and the filtrate to the RIT/Orbitrap inlet.

The system shown in Figure 4 adequately demonstrated the various functions however it had several limitations that need to be modified prior to development of a flight instrument. These disadvantages included:

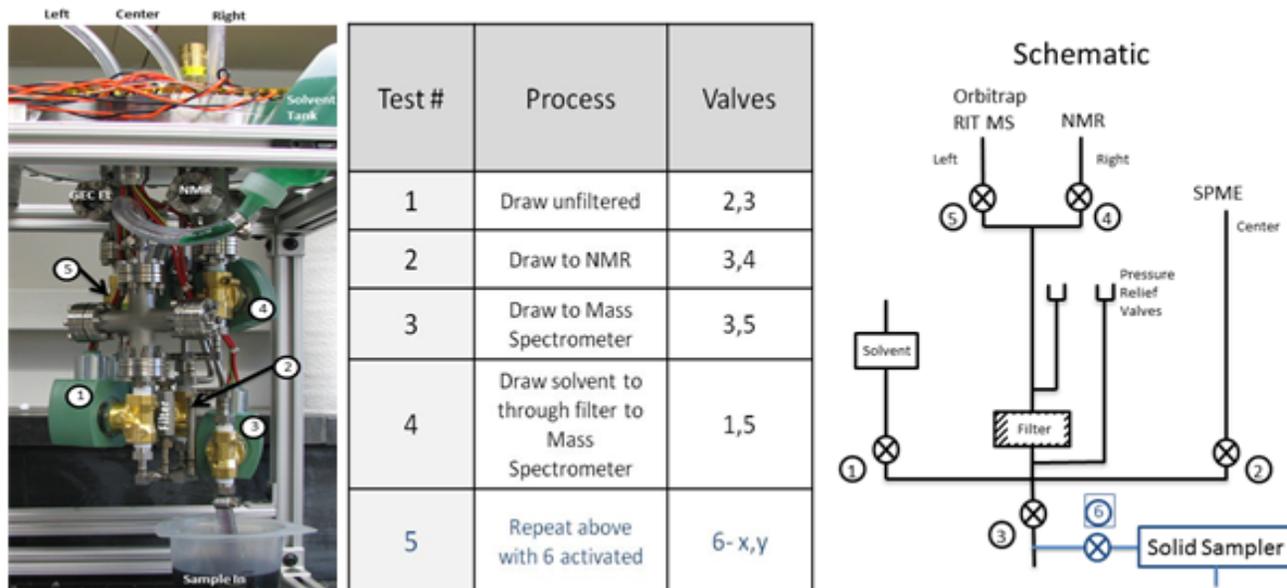
1. The system and mass were too large for a potential flight implementation
2. Though the valves could pump cryogenic fluids they can not be submersed.
3. The power required to activate the valves was too large for a potential flight instrument.

In order to develop the above system into a flight compatible design we investigated the development and design of low mass/volume low power cryogenic valves for space. A variety of materials and implementations were considered including low temperature Shape Memory Alloys, cryogenic solenoid valves and the best candidate actuator material for this application based on mass and power and reliability was the new single crystal materials that have been developed for cryogenic applications.

## III. PIEZOELECTRIC ACTUATION AT CRYOGENIC TEMPERATURES

Piezoelectric materials convert applied electrical signals into a displacement which can be used and amplified to produce strokes up to 1 mm. For a piezoelectric material the stroke per volt is represented by the piezoelectric charge coefficient  $d_{33}$  which has units of C/N or m/V. For example PZT 5H a soft piezoelectric ceramic material used in medical transducers has a nominal  $d_{33}$  coefficient of  $580 \times 10^{-12}$  m/V. Currently, the majority of piezoelectric materials for such transducers are ferroelectric materials due to their high electromechanical properties, which arise from the two types of contributions; the

intrinsic (lattice effects) and extrinsic contributions (the motion of domain walls) in ferroelectric materials.



**Figure 4:** Bench top sampling system and schematic diagram of the valving with an activation sequence chart.

One of the most important characteristics of this kind of material is the morphotropic phase boundary (MPB), which refers to the boundary between two compositions where the two phases are present in equivalent energy states. MPB is an important concept for ferroelectric materials as MPB compositions offer enormously high dielectric and piezoelectric properties as a result of enhanced intrinsic contributions. Lead Zirconate Titanate (PZT) is one of the most widely used piezoelectric materials because of its excellent piezoelectric and dielectric properties near the MPB.

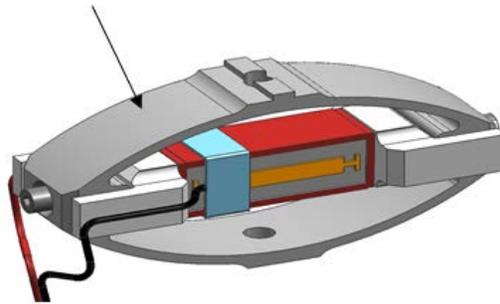
The piezoelectric response contains not only the intrinsic contribution, but also an extrinsic contribution caused by movement of non-180° domain walls, which is strongly temperature dependent. MPB-based PZTs are generally tailored with dopant ions, which impede or facilitate domain wall movements. Importantly, in PZT ceramics, more than 50% of the net piezoelectric responses arises from these extrinsic contributions<sup>8</sup>; therefore, when PZT materials are used at cryogenic temperatures, most of the extrinsic contributions are frozen out, consequently, the materials lose their piezoelectric performance; for example, the piezoelectric  $d$  coefficient was reported to decrease from 760 pC/N to 220 pC/N when the operating temperature was decreased from 300K to 30K. This decrease indicates the necessity for appropriate piezoelectric materials to be used to make an appropriate actuator for operation at cryogenic temperatures

Recently, domain engineered <001> relaxor Lead Titanate (PT) single crystal family, such as PZN-PT, PMN-PT and PIN-PMN-PT, has been studied extensively due to their extremely high piezoelectric responses, strain over 1.7%, piezoelectric constant  $d_{33}$  over 2000 pC/N, electromechanical coupling factor  $k_{33}$  over 90%, with almost non-hysteretic strain-field behavior. Since the origin of such high electromechanical properties of relaxor-PT single crystals is due to the polarization rotation effect<sup>9,10</sup>, (i.e., intrinsic contributions), the property degradation at cryogenic temperatures is much lower than in PZT ceramics, making them promising candidates for cryogenic actuators from the perspective of stroke and power loss in the actuator when activated.

In this valve design, the relaxor-PT single crystal transducers, specifically TRS Inc. (PIN-PMN-PT)  $d_{32}$  bars as the active layer, will be incorporated into flextensional frame and used to as the valve actuator. A solid model of a flextensional actuator which has been tested for Mars conditions<sup>11</sup> is shown in Figure

5. Three of these actuators shake the inlet funnel of the CHEMIN instrument on Mars Opportunity rover currently on Mars. Piezoelectric materials can exert a large force however the strain is limited by the field across the piezoelectric and the piezoelectric constant. Flextensional actuators are stroke amplifying devices. They operate as levers in that they increase the displacement at the expense of the delivered force. These actuators allow for the Force and Stroke to be tailored for a specific application by adjusting the angle  $\theta$  of the flextensional frame with the horizontal axis. The stroke amplification<sup>12</sup>  $A$  and force reduction  $A^{-1}$  are defined by  $A = -\cot(\theta)$ .

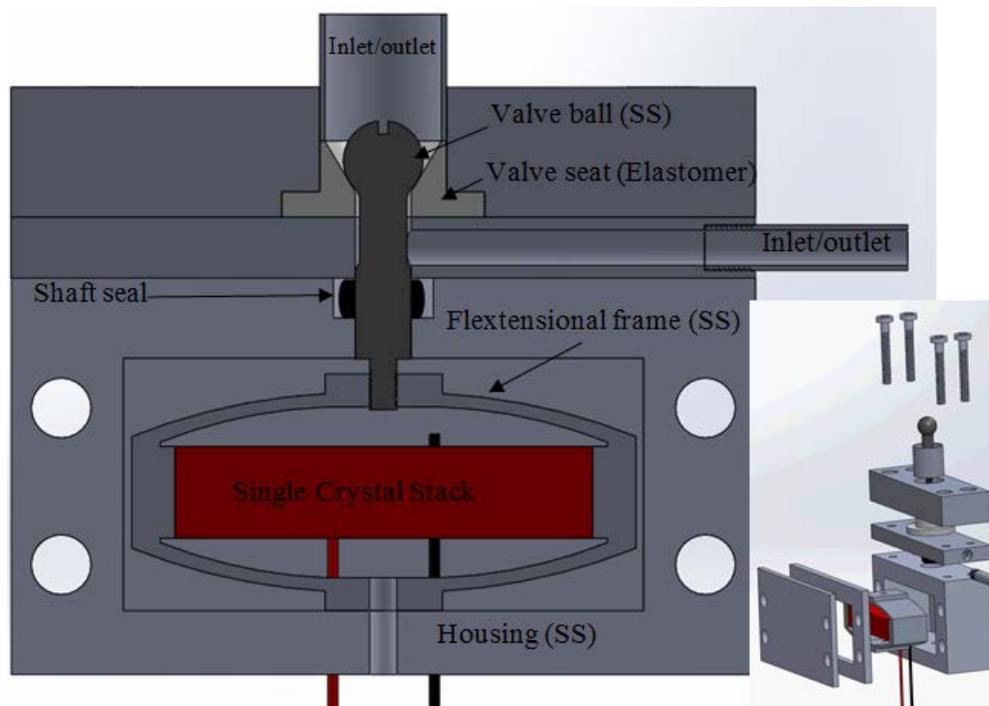
**Flextensional Frame**



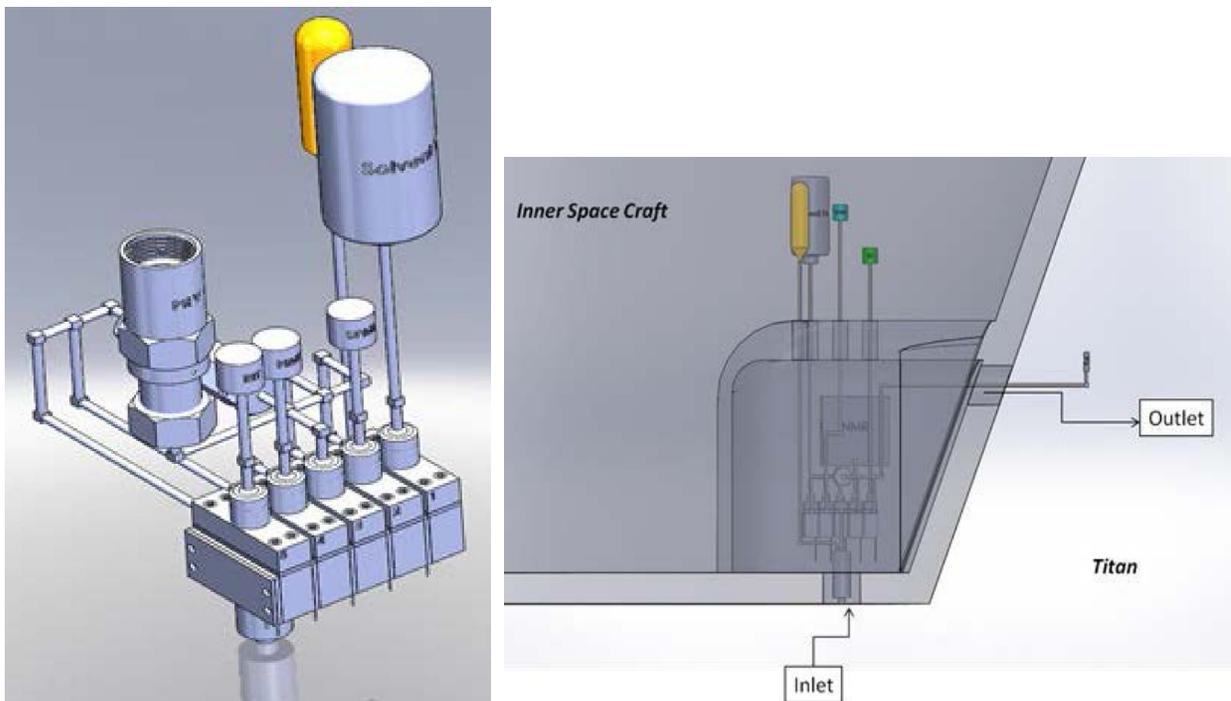
**Figure 5:** Example of flextensional actuator designed for the CHEMIN Funnels [Sherrit et al.<sup>11</sup>]. Piezoelectric stack is red. Major Actuation is on top and bottom face of flexure.

In order to design a system with the mass, volume and power envelope appropriate for a potential flight design we have incorporated flextensional transducers produced by TRS Technologies Inc. The actuator dimensions for this flextensional design have a 25 mm x 12 mm x 6 mm volume space. The actuator mass is 7.9 grams. A cross sectional diagram of the valve including the actuators chosen for the cryogenic valve are shown in Figure 8 along with a detail of the valve assembly. A variety of suitable cryogenic elastomer materials (Torlon, Viton, Teflon) at cryogenic temperatures can be used to seal and various valve seating geometries e.g., ball and conical seating seal to determine the most reliable seal configuration.

The material of the flextensional frame (SS 15-5PH H900) and the housing is designed to be the same material to minimize thermal stress in the materials. The valve will be assembled at room temperature and activated at 90 K. This 180 K temperature delta can cause significant differential thermal expansion. Since the flexure is a soft spring, the overall effect on the pre-stress on the piezoelectric should be minimal. The DC power dissipation in the actuator is of the order of milliwatts. The miniature system including the filter, pressure relief valves and solvent tanks is shown in Figure 6. The overall sampling system shown in Figure 7 has five independent valves which when combined have the same functionality as the bench top system shown in Figure 4. The front end of the sampling system contains a pneumatic penetrator for solid sampling in addition to the liquid sample inlet tube. The pressure relief valves and filters are off the shelf and will be purchased by Lee Products Inc<sup>13</sup>. The valve shown in Figure 6 is normally closed. The valve ball is set in the flextensional frame near the operational temperature of 100 K. An O-ring around the shaft of the valve ball seals the inlet and outlet from the chamber housing the flextensional actuator. The PIN-PMN-PT  $d_{32}$  bars actuator poling was done at 5 kV/cm or 950V. The nominal displacement for an applied voltage of 200V is 600 microns. The piezoelectric stacks without the flextensional frame were measured to have a stroke of about 111 microns per 100 Volts. Typical decreases of the piezoelectric stroke from room temperature to 100 K are about 40%. The piezoelectric crystal has a blocking stress of 20 MPa. The crystal is 5 mm x 5 mm so the blocking force is about 500 N however the flextensional has an amplification factor  $A = 300/111 = 2.7$  so on the face of the flextensional the blocking force is about 180N. The force required to open against a 1.5 bar pressure on the valve stem is about 1-2 N ignoring friction of the O-ring. The free-free impedance resonance



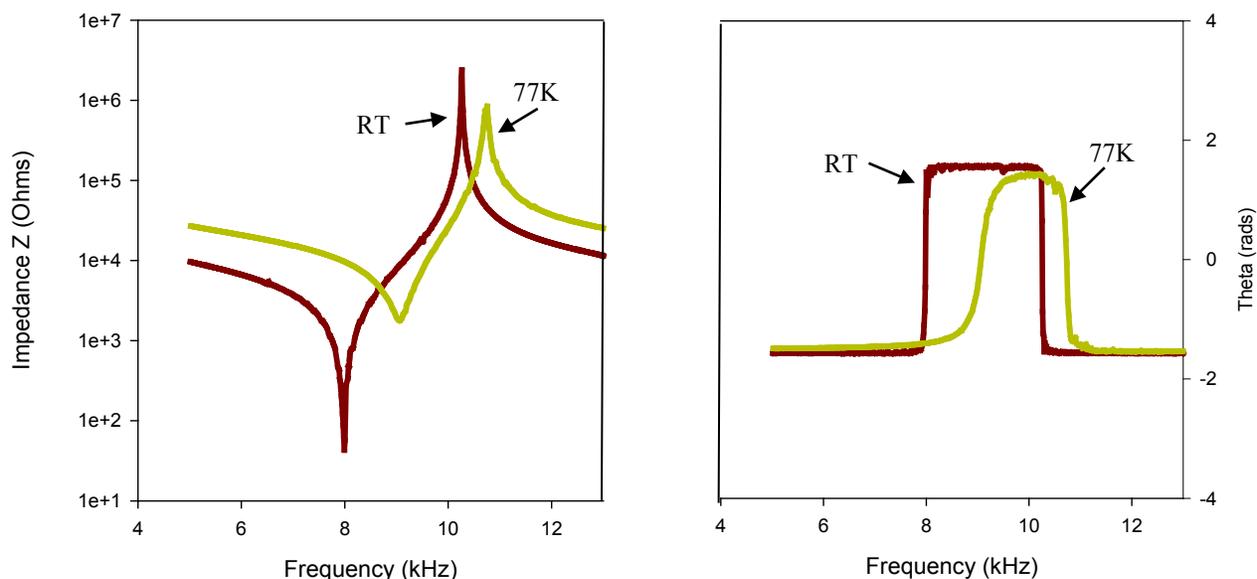
**Figure 6:** Cross section of the CAD model of the cryogenic actuator along with an assembly blow up.



**Figure 7:** Miniature sample and sample handling system that is being developed and its position with respect to the spacecraft. The yellow tank is liquid nitrogen which is used to drive the solid penetrator. The small cylinders above the valve assembly are input ports to instruments. The solvent tanks and the pressure relief valve are marked.



**Figure 8.** A photograph of the PIN-PMN-PT  $d_{32}$  flextensional actuator.



**Figure 9.** The free-free impedance resonance spectrum for the flextensional is shown in Figure 8. at room temperature and liquid Nitrogen temperatures. The sharper, lower frequency resonance is the room temperature curve.

spectra for the flextensional shown in Figure 8 is plotted in Figure 9 at room temperature and liquid Nitrogen temperatures. At room temperature the electromechanical coupling is  $k = 0.65$  while the resonance frequency is 7.71 kHz and the antiresonance frequency is 9.73 kHz. The capacitance is 2.7 nF. At liquid  $N_2$  temperature the electromechanical coupling is  $k = 0.57$  while the resonance frequency is 9.081 kHz and the antiresonance frequency is 10.74 kHz. The capacitance is 1.07 nF. The leakage current for an applied voltage of 21 Volts ranges from 3.5  $\mu\text{A}$  at liquid  $N_2$  temperatures to 5  $\mu\text{A}$  at room temperature. The power dissipated at 200 Volt based on a linear extrapolation is therefore in the range of 6.7 mW to 9.5 mW which is about 3 orders of magnitude smaller than the cryogenic valves shown in Figure 4. The percent of the room temperature stroke that is available at 77K based on the effective piezoelectric coefficient determined from the resonance is approximately 46% or about 280 microns based on the nominal stroke from the manufacturer specifications.

## IV. CONCLUSIONS

We have described a sampler that can be used in extremely cold environments to sample and distribute cryogenic fluids to a variety of instruments. The sampler and sample distribution system was designed for one of the potential future mission concepts to explore Titan with a floating Lander on a Titan Lake and determine the local lake chemistry. In order to accomplish this within the expected mass volume and power budgets we have pursued the development of low power lightweight cryogenic valves which can be used with a vacuum system to sample lake liquid and to distribute the sample to various instruments aboard the Lander. To meet this need we have chose to develop low power cryogenic valves and actuators based on a single crystal piezoelectric flextensional stacks produced by TRS ceramics Inc. Since the origin of such high electromechanical properties of Relaxor-PT single crystals is due to the polarization rotation effect, (i.e., intrinsic contributions), the strain per volt decrease at cryogenic temperatures is much lower than in standard Lead Zirconate Titanate (PZT) ceramics. This makes them promising candidates for cryogenic actuators with regards to the stroke for a given voltage. The initial testing of the actuators showed about a 54% decrease in the small signal effective piezoelectric constant from room temperature. The fine design details including incorporating the stroke field relationship in the valve for optimal performance is currently being developed.

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