PRELIMINARY CRYOVISCOMETRY OF METHANOL-WATER AS A TITAN CRYOMAGMA ANALOGUE. K. L. Mitchell[1] (Karl.L.Mitchell@jpl.nasa.gov), F. Zhong[1], C. C. Hays[1], M. Barmatz[1], R. Hodyss[1], J. C. Castillo[1] and L. E. Robshaw[2], [1]Jet Propulsion Laboratory, Mail Stop 183-601, 4800 Oak Grove Dr., Pasadena, CA 91109-8099, USA, [2]Lancaster University, Environmental Science Dept., Lancaster LA1 4YQ, UK.

Introduction: In order to improve our ability to model the emplacement of cryovolcanic flows and domes on Titan (e.g. Lopes et al., 2007) and other bodies, we have embarked on a series of experiments to measure the rheological properties of cryovolcanic materials and analogues, based on earlier work by Kargel et al. (1991) but with improvements to thermal control and explored parameter space.

Our initial experiments, with methanol-water, are primarily for calibration of our system – we do not propose that cryomagmas on Titan are methanol-water – but should also yield insights into slurry dynamics in general, and may be of relevance to other outer solar-system bodies.

Apparatus: We have constructed a cryogenic rotational rheometric system (fig. 1) based around a Brookfield Engineering HBDV-III Ultra rheometer. The spring torque for this model is 5.7496×10^{-3} N m, with speed ranges from 0.01 to 250 RPM, capable of measuring viscosity in the range 80 Pa s to 6.4×10^{6} Pa's when several spindles are used over many speeds. All system components in contact with the cryogens are made from stainless steel 316 (marine-grade), unless otherwise noted, to minimize chemical interactions especially with ammonia-based slurries which will eventually be studied. Thermal control is achieved by a combination of a liquid nitrogen bath and a series of calibrated diode thermometers and copper heaters, controlled from a LabVIEW-based computer system.

Sample cell. The cryogenic slurry is placed into a sample cell of 8.25 cm-diameter, 12 cm-height. The cap is sealed with indium wire to the flange, and the tubular conduit for the spindle is silver-soldered to an adaptor, sealed to the cell cap center with an indium wire. A calibrated diode thermometer and a heater are mounted on the outer surface of both the bottom plate and the cap, each of which can be independently regulated to any temperature from -133 C to 20 C. Heaters and thermometers are located in order to minimize the horizontal temperature gradient on the bottom plate and the cap. The sidewall is wrapped with a fifteen-layer Multi-Layer-Insulator (alternate aluminum and Mylar sheets) to reduce the uncontrolled radiation heat input to the sample from the sidewall.

Vacuum Can. The sample cell (aluminum 6061-T6) is fitted into a vacuum can of 12 cm diameter and 16.8 cm height. A 0.635 cm thick aluminum cap is sealed to the can with an indium wire. A brass adaptor is silver-soldered to the conduit and sealed to the center of the vacuum can cap with an indium wire; thus, the relative position between the cell and the vacuum can is fixed. There are four additional ports on the vacuum can cap beside the one for the conduit. One of the four ports is used for both pumping out the can and the conduit of the electrical wires, with the remainder ports capped for future additional features. The sample cell takes the most cooling power via the radiation at the top and bottom. The spindle conduit acts as an additional thermal link between the cell top and vacuum can top, and a stainless steel shim stock of 0.01 cm-thickness, warped as a springy thermal contact between the cell bottom and vacuum can bottom, is employed to slightly improve cooling efficiency.

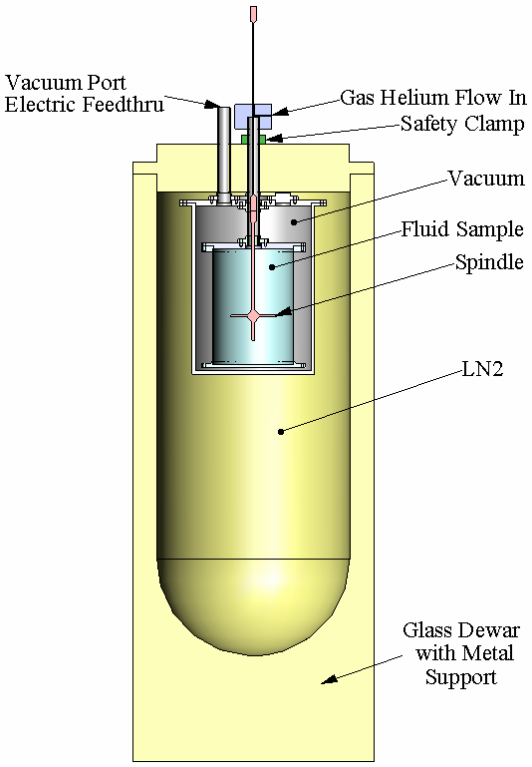

Fig. 1: Experimental schematic.

Dewar. The vacuum can is placed into a Finemech 20 cm-diameter (internal), 50 cm-deep, 14-liter KGW-1243 cylindrical dewar flask, suspended from the dewar cap by a clamp. The custom-ordered dewar cap has a reinforced stainless steel plate from which the vacuum can is suspended. The dewar is filled with

12.8 litres of liquid nitrogen, which is typically consumed in ~140 hours.

To protect the fluid sample from air condensation via the center conduit, an argon gas flow was connected to the conduit opening to establish a "pressure cap". There is no need to submerge the entire vacuum can into liquid nitrogen since the rheology measurements were to cover temperature only down to 150 K. Copper foil strips are attached to the vacuum can and reached the dewar bottom, connecting the can to the cooling reservoir and optimizing longevity of the cooling time.

Method: A 40% mixture by weight of methanol in ultra pure water was prepared, both components having been de-gassed. To allow for possible thermal expansion of water during solidification, only 500 ml fluid sample was filled in the 600 ml sample cell.

The #2 spindle, used most in the current cryo-ice experiment, ranges from 80 Pa's to 6.4×10^4 Pa's with 0.5 to 100 RPM. This spindle has a disk of 4.7 cm diameter and 0.16 cm thickness.

The first experiment was to check for strain-rate dependence of the slurry, so measurements were made at 5 rotation rates, from 6.25 to 100 RPM, at contant *T*.

Subsequent runs were at fixed rotation rates and over varying temperatures (-40 to -100 C) in 1 degree intervals. A LabVIEW routine was written to check, in real-time, whether the cell bottom temperature has reached equilibrium. Typically this takes ~ 20 minutes for each temperature step.

Results: Strain rate dependence was clearly observed in the intial experiments, as shown in Fig. 2 (top). This strain rate dependence will be checked with freshly made samples.

Temperature-dependence of viscosity was also clearly observed, e.g. Fig. 2 (bottom). The measurements above the transition temperature (-45 C) were too noisy for the rates lower than 20 rpm; such torques barely registering above the instrument's baseline noise. When the torque reached the full scale at a temperature, the rotation rate was decreased by half in order to have the torque fall back to within its range. This procedure was repeated until the rotation rate reached 0.5 RPM at which the measured viscosity showed significant oscillation of the same frequency. It was suspected that such oscillation was caused by the extension that used hooks to link between the drive and spindle. Future additional temperature dependent viscosity runs will be performed to validate reproducibility.

Summary: The rheology experiment setup was completed and preliminary results are satisfactory to first order, although will require refinement in order to make them truly reproducible. We will improve the system by: (1) Enabling refilling of the liquid nitrogen in situ; (2) Using a new extension rod for greater stability; (3) Inserting an interior thermometer along the rotation axis from the cell bottom for improved temperature control; (4) Adding an optical window and LED light source to measure light intensity and hence yield information on solidification, and possibly allow limited direct observations of the slurry.

Routine operations will commence, as soon as the system is optimized, with ammonia-water being the first major material of interest. These experiments will be accompanied by Differential Scanning Calorimeter measurements at similar cooling rates in order to determine appropriate phase diagrams and explore non-equilbrium phase behaviour.

References: [1] Lopes R.M.C. et al. (2007) *Icarus* **186**, 395-412, doi:10.1016/j.icarus.2006.09.006. [2] Kargel J.S. et al. (1991) *Icarus* **94**, 368-390.

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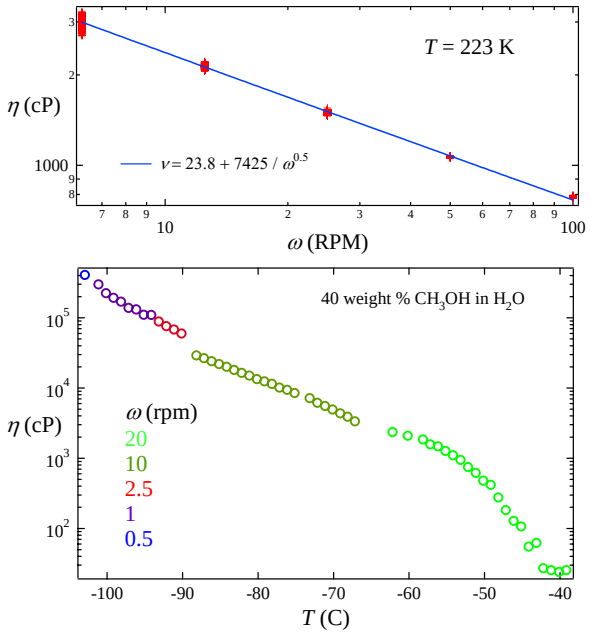

Fig. 2: Results for a 40% methanol in water mixture. (top) Demonstrating strain-rate-dependence of viscosity. (bottom) Demonstrating temperature-dependence of viscosity.