Making a Propane Clathrate Hydrate

USGS.gov

Janda Lab, UCI

By: Kathy Marvin

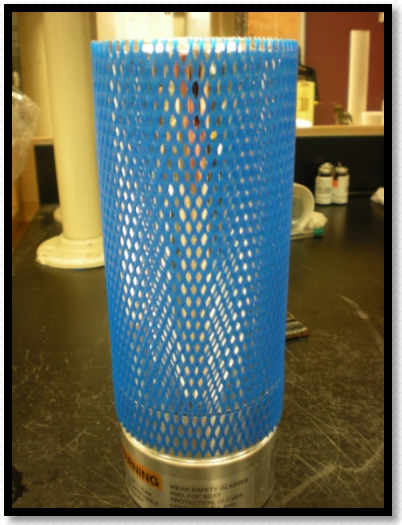
Summer, 2010

Materials

- 4L liquid nitrogen in dewar
- 500 mL dewar flask
- 65 mL nano pure water (purified to a nano scale, meaning that all particles in this water are smaller than a nanometer (µm), or one billionth of a meter)
- 25mL buret with stopcock attached to stand
- Strainer
- Coffee grinder (cooled in freezer)
- 180 µm sieve with fitted pan
- Wooden cylinder (used for grinding, kept in freezer)
- Stainless steel pressure chamber (cell), with temperature probes
- Hex wrench to fit screws on cylinder top
- Propane

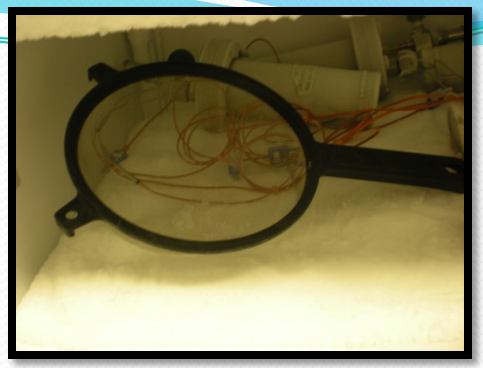
- 4L liquid nitrogen in dewar

- 500 mL dewar flask



- 25mL buret with stopcock attached to stand

- Strainer (cooled in freezer)



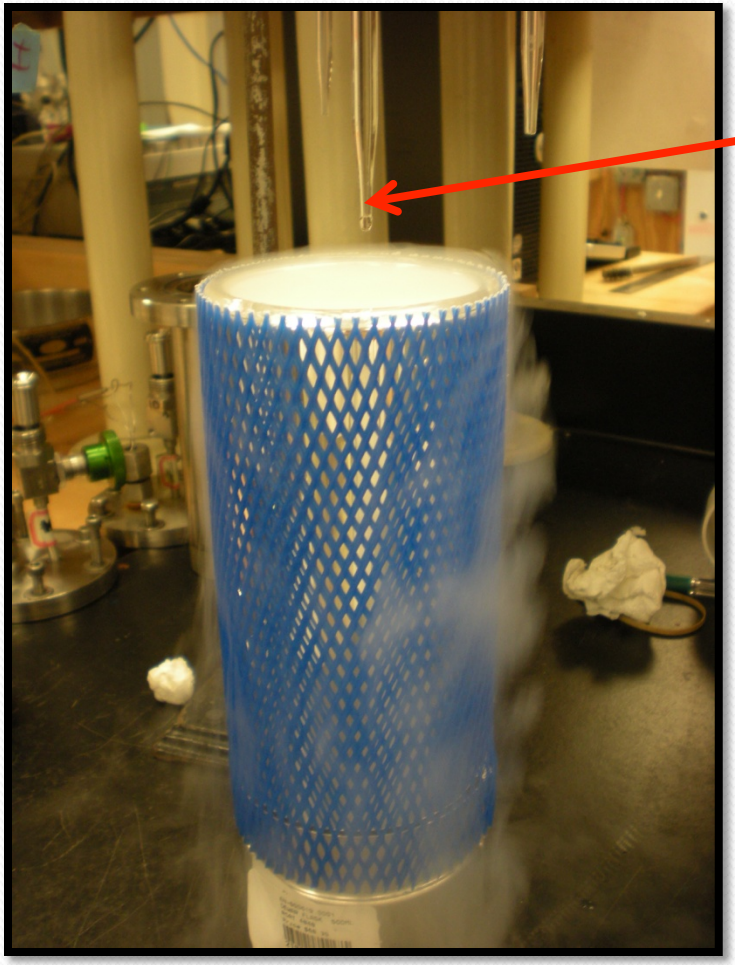
- Coffee grinder (cooled in freezer)

- Wooden cylinder (used for grinding, cooled in freezer)

•180 µm sieve with fitted pan

•Stainless steel pressure chamber (cell), with temperature probes

- Fill dewar flask with liquid nitrogen and place it below the buret



Tip of buret with cockstop above

- Fill a buret with nano pure H_2O
- Drip the nanopure water into the dewar flask
- Set the stopcock so that drips of H_2O are individual but dripping as fast as possible
- Drip 65mL of nanopure H_2O into the liquid nitrogen

- Use frozen strainer to separate ice pellets from liquid nitrogen; reserve strained liquid nitrogen
- Break up ice pellets that are ‘stuck’ together

- Using the coffee grinder in the freezer, grind the nano pure ice pellets on finest grind to make ‘snow’
- Place the ground ‘snow’ in freezer for a few minutes to maintain temperature

- Pour liquid nitrogen to a depth of about 1cm, into lower pan through top sieve to cool both pieces. (You will have to wait while it boils and add more until the temperature stabilizes and the liquid nitrogen remains in the pan)

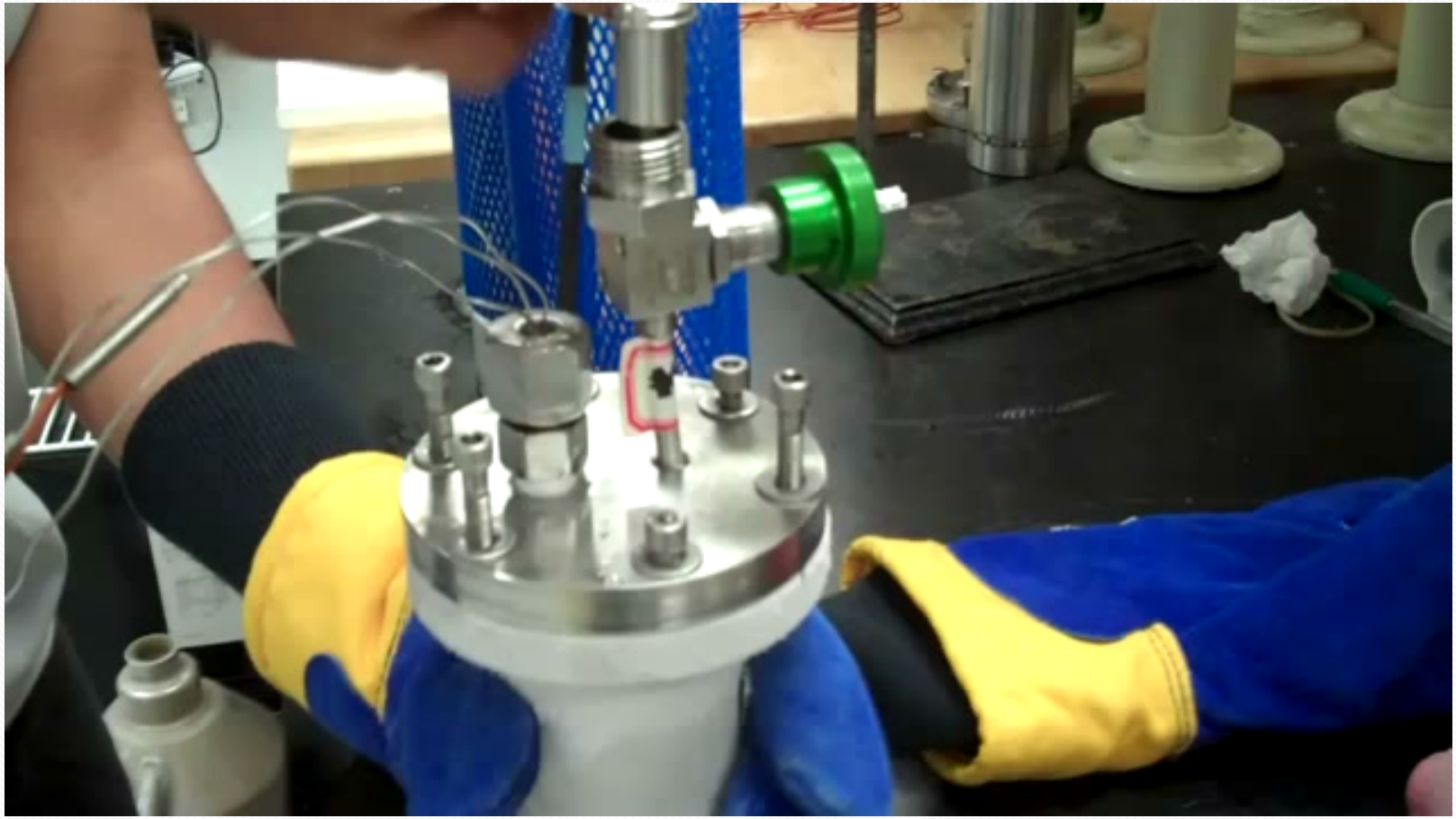
- Pour the ‘snow’ from the ground ice pellets into the top sieve
- Using the frozen wooden cylinder, grind the snow through the screen creating tiny 180µm particles to fall through to the liquid nitrogen below that will keep them individual and stable throughout this process

- Grind the snow to 180μm because increasing the surface area of the ice by creating smaller pellets of snow will allow for more areas for propane to enter the water cages and a greater amount of clathrate hydrate to be formed.
- Example: If you cut a loaf of bread into slices, each time you cut a new slice, you get an extra surface onto which you can spread butter . The thinner you cut the slices, the more slices you get and so the more butter you can put on the bread. By chewing your food you increase the surface area so that digestion can go faster.
- Studies on clathrate hydrates show that decreasing the size of the ice pellets to 180μ allows for increased penetration of the C_3H_8 into the gas hydrate

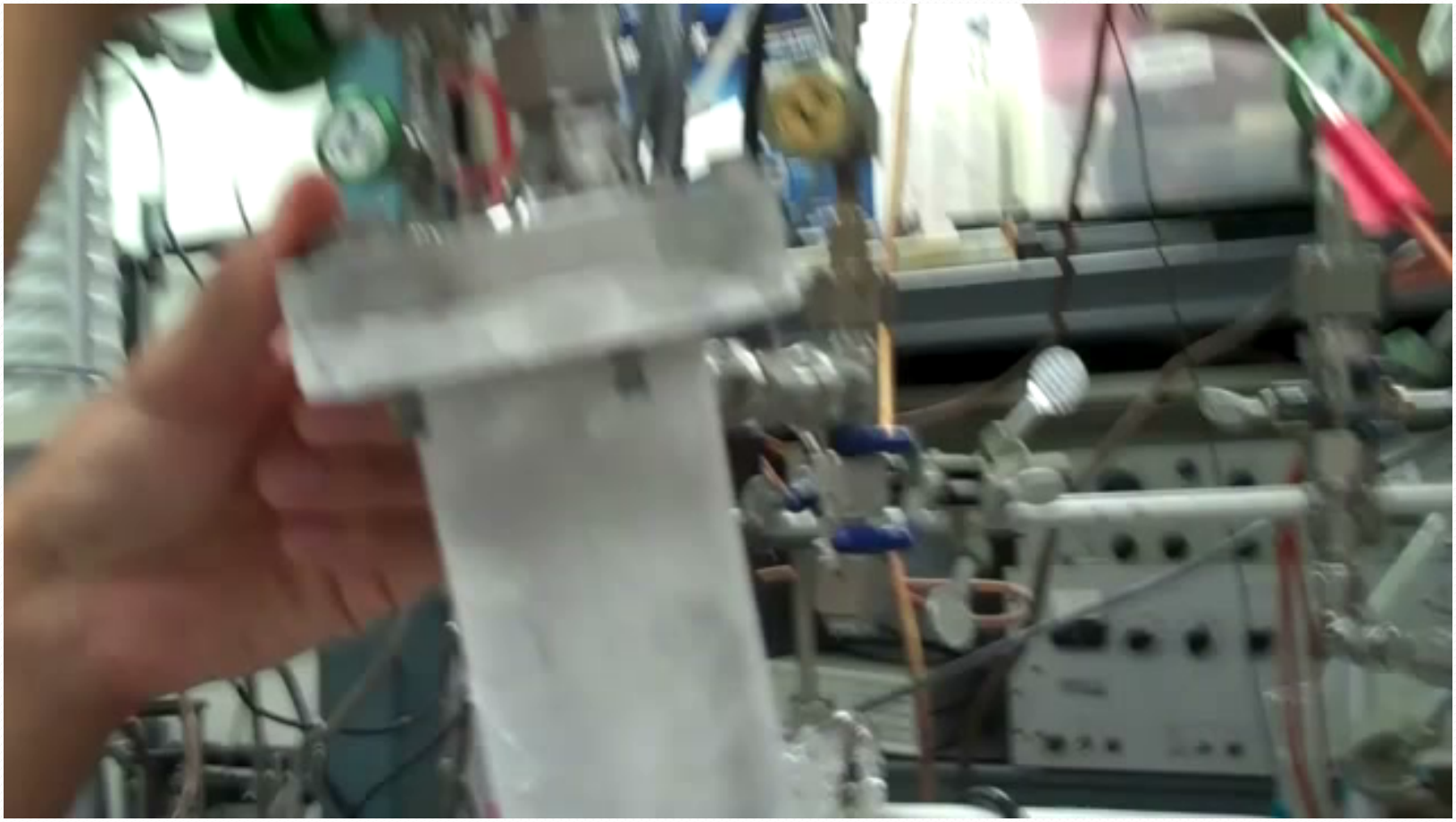
- Check the level of liquid nitrogen a few times to make sure the fine ‘snow’ is staying frozen. Add a little more liquid nitrogen when necessary
- Occasionally, dip the wooden cylinder into the liquid nitrogen in the dewar flask to keep the grinding end very cold for grinding
- Stop when most of the ground ice is sieved
- Weigh 31g of snow into a frozen container or small Styrofoam cup
- Pour measured snow into pressurized cell

- Place top on cell with temperature probes inserted into center

- Place top on cell and secure all screws so that no propane can escape.
- Use hex wrench to tighten all screws securely.



- Place pressure cell into ethylene glycol water bath set to a minimum of -3°C and tighten all screws. Make certain pressure valve is closed



- Bleed all air from lines to pressure cell
- Open propane tank and set to desired psi
- Open needle valve to allow C_3H_8 to enter pressure cell slowly
- Check computer readings to make sure temperature inside pressure cell remains at least -3° C so that snow will not melt
- Wait for 2-3 days, allowing time for gas propane to become trapped inside H_2O molecules
- After 2-3 days, close all valves and turn off incoming propane gas

- Slowly release pressure from within the cell which will cause the cell to drop in temperature quickly as trapped propane is released. (this should be done in a fume hood or outdoors)
- Keep pressure cell in freezer or on ice until ready to test it.
- When ready to light sample of clathrate hydrate, open all screws on the pressure cell with hex wrench.
- Remove frozen ice sample which now has C_3H_8 trapped inside the water crystals
- Place sample (which looks very much like a white candle) on a flame proof dish or screen
- Ignite with match or small torch
- Propane clathrate hydrate will burn as the ice melts

- Sample #1 of a propane clathrate hydrate made in the Janda Lab at UCI, July 2010, burning.

- Sample #2 of a propane clathrate hydrate made in the Janda Lab at UCI, July 2010, burning inside a fume hood.