Karl Martinez's BZ Manual – 07 Feb 2005

Contents:

I. Tips on Running the BZ Experiment

A. Chemistry
1. Preparation
2. Materials & Safety

B. Experimental Hardware
1. Vycor
2. P500 Pumps
3. Tubing and Reactor
4. Imaging Light
5. Image Acquisition
6. Projector

C. Old Linux-based System
1. Data format

D. Photodiode Power Measurements

II. Experimental Observations

A. On Excitability
1. How BZ responds to inhibitor concentration
2. Tongue bending & other tricks

B. Spatial & Stochastic Forcing
1. Preliminary results
2. Labview instructions

I. Tips on Running the BZ Experiment

My experience is in running the continuously stirred, two feed, open reactor with vycor as the reaction medium. There are a number of components which need to work properly in order to observe oscillations/spirals and for there to be a robust response to the optical forcing.

You should go to Professor Lin and anyone else with extensive experience if you have questions. If something I've written needs clarification, send me an email and I will try to reply promptly.

A. Chemistry

1. Preparation

The most important thing to remember with the chemistry is to be consistent each time you make a solution. Basically, make the solutions the same way each time, unless you find something that needs to be changed. Try to use the same brand and make of sulfuric acid, potassium bromate etc. and prepare the stock solutions the same way each time.

Our methods of preparation so far have been adequate for observing pattern formation from the resonant forcing. Our standard stock recipes are posted above the scale in the hood room.

The basic procedure for making a solution: example:1L of 0.23M KBrO3, 1M H2SO4 in water

Start with a 1L volumetric flask. Note the line etched into the neck of the flask denotes 1 Liter. Fill the flask about half way with distilled water. Drop a stir bar into the water in the flask and place the flask on top of a magnetic stirrer. The stirrer mixes well at any setting below 5.

Then, using a graduated cylinder and a funnel in the mouth of the volumetric flask add 200 mL of 5M H2SO4 to the water. As a matter of safety always add acids to distilled water, not water to acid. The acid will mix quickly.

Now weigh out 38.41g of solid KBrO3 onto wax paper on the scale. You can use a steel spatula to handle the solid potassium bromate. Then, with a wide mouth funnel in the flask dump the solid KBrO3 into the flask. If some solid gets stuck in the funnel use distilled water in the water bottle to wash the solid down into the flask. Fill the rest of the flask with distilled water until about two inches below the 1L liter line. The solid takes about twenty to thirty minutes to completely dissolve.

Use a magnet to pull the magnetic stirbar above the fill line in the volumetric flask. Then you can dilute the solution to the fill line and drop the stirbar back in to finish stirring. Of course, wait for the solution to finish disintegrating in water before filling to the line, as some solutions may expand or contract upon dissolution.

Label the solution with the concentration of the chemicals, date and your name and add it to your experiment.

The malonic acid-sodium bromide solution and the ruthenium solution are made in a similar manner. Just follow the posted recipes.

Sulfuric Acid stock:

When making the 5M sulfuric acid stock start with a 2L volumetric flask with at least a liter of distilled water. When you add the highly concentrated sulfuric acid from the manufacturer stir for only a few seconds and then place the flask in a bucket with lots of ice. You should have at least a bucket full available. See the safety section for more info. Give this solution time to cool before you fill it to the line.

Malonic Acid Stock:

After adding the solid malonic acid to the distilled water, let it dissolve for several hours before filling it to the line. Then filter the solution with an aspirator-Buchner funnel set up using filter paper with the pore size of VWR brand 28321-135(in the lab right now) or smaller. A cloudy malonic acid stock solution will yield chemistry which doesn't react strongly to the light. You have to remove the impurities with the small pore size.

2. Materials & Safety

Again – Always add acids to water, not water to acid.

Keep the solid ruthenium away from light. The ruthenium is photosensitive (hence we can force the reaction with light), but the photosensitivity declines with long exposures to light. Limit Ruthenium's exposure to light.

Sulfuric Acid stock:

When diluting the sulfuric acid to 5M solution use lots and lots of ice, at least a bucket full. The reaction is highly exothermic and dangerous if not properly cooled. Also, wear full protective gear, gloves, goggles and a lab coat, and work under the hood when handling the concentrated sulfuric acid.

B. Experimental Hardware

1. Vycor

Just in case it hasn't been emphasized enough the porous Vycor glass is the most critical and fragile piece of equipment used in this experiment. Handle Vycor with the utmost care (latex gloves, and plastic tweezers are best for handling). It breaks easily and any dirt or silicone which gets into the glass can ruin it for use in experiments. Porous Vycor is expensive and very hard to get.

We use Silicone to attach the Vycor glass to the plexiglass holder. I suggest applying a liberal amount of silicone to the lip of plexiglass where the Vycor will sit. Make sure there are no bubbles and using the straight edge of a spatula to remove excess silicone from the holder. In my experience if you run a spatula about the indention where the Vycor fits with the spatula touching the top and bottom edge of the holder (forming a triangle) it should leave the right amount of silicone to hold the Vycor without being excessive. After gently placing the Vycor on the holder put a Kimwipe, rubber stopper and 500g weight on top to press it down. Let it sit for a full 24 hours so the silicone will dry.

For new, unused membranes, when first placed in the reactor with the BZ chemicals it takes 2 to 6 hours before we observe spiral patterns or oscillations of any kind. Initially a lot of bubbles will form on the membrane. After running for a couple of days most of the bubbles should go away.

Realize, leaks 'through' or 'around' the membrane are fairly common. In fact if you get spiral patterns, but the reaction doesn't respond to high intensity forcing light, more than likely you have a membrane problem. If working properly(no leaks) the malonic acid side of the reactor will be colorless and clear. The Ru side of the reactor will be a light green(ask Anna about this, I'm red green colorblind). If you think there is a leak simply add a second plexiglass holder to the reactor so that it covers the edge portion of the membrane.

Membrane removal:

Use xylene to remove the membrane from the plexiglass holder. This takes patience. It is very easy to get plexiglass or other substances into the membrane, ruining the membrane.

What I suggest is place the membrane and holder flat onto a 100mL beaker with the flat side facing down. Using a pipet put a few drops of xylene onto the side of the membrane facing up. Let it sit for about a minute and then remove it using water sprayed from a squeeze bottle. Add more xylene and repeat until the membrane becomes partially unglued from the plexiglass. Notice while you do this some of the plexiglass will come off and float in the xylene. So it doesn't get on the membrane remove the floating plexiglass using tweezers.

The partially unglued membrane can be gently removed from the holder using tweezers. The membrane will probably still have silicone around the edge. Bath the membrane in a petri dish full of xylene. The silicone will start to come off the membrane. Using 2 pairs of tweezers gently pry the silicone off of the membrane. Once the silicone is removed bath the xylene in ethyl alcohol for about an hour. The alcohol removes any excess xylene which might be in the membrane. Xylene in the membrane will show up as dark patches with low oscillation frequencies.

2. P500 Pumps

The repair manuals for the P500 Pumps offer good instructions on basic maintenance and repair. The pumps with sulfuric acid should be checked regularly (once per month) as any leaks will corrode the piston rods which are expensive to replace. I suggest cleaning out all the pumps and testing their flow rates at least one time per year.

3. Tubing and Reactor

Tubing, premixers and the Reactor do become clogged with salts. If you see a leak at a particular joint in the various plumbing that doesn't necessarily mean the problem is at that joint, usually it is somewhere further along in the plumbing, like the reactor.

An easy troubleshooting method is simply remove the joint at the next connector downstream from the leak, but keep pumping like normal. If there is still a leak at the same joint then there is your blockage. If not connect the next piece and the next until you see the leak. When you see the leak you have found the blocked piece (often a dalron tubing screw).

4. Imaging Light

The frosted glass placed in front of the reactor is very important. There are a number of scratches and imperfections in the reactor windows which are magnified without the frosted glass to scatter the incoming light.

Note: the imaging light isn't spatially homogeneous. The difference of intensity can vary significantly from one region to another. Try different orientations of the imaging light to get the best image.

Also there is a give and take between imaging light intensity and the aperture size/fstop on the camera lens. Open the aperture all the way and you can minimize the light intensity of the imaging light. See the section on image acquisition for more information.

5. Image Acquisition

There is significant glare from the reactor face when the projector light is on to force the reaction. This is an annoying feature we can deal with two ways. First, you can tilt the reactor backward or forward about 10 degrees. The tilted reactor reflects the projector light at an angle preventing it from getting into the camera. Your second option is to significantly close the camera aperture. This will limit the amount of glare which gets to the CCD chip, but you will have to increase the imaging light intensity in order to see an image of the reaction.

A narrow bandpass filter (450nm +-10nm) is taped to the camera lens. The features of the reaction we want to see (Ruthenium concentration) are best seen at the absorption peak of approx 452nm. Without the filter we just see a bright light streaming through the membrane.

6. Projector

Also, the ruthenium side of the reactor should always be away from the projector light

C. Old Linux-based System

The old linux box, Couette has an array of C programs we used to run the experiment from 1995 to mid 2002. The data Format is distinct from what we used from Fall 2002 until the present time. The data is stored as 256X240 XXXXX bit images.

D. Photodiode Power Measurements

The light intensity from the projector bulb should be checked at least once every time it is turned on. This can be done using the Coherent Powermeter with the LV detector head. Set the Powermeter to measure at 450nm, this will best calibrate the Powermeters chip measurement. Also, use a broadband light filter 450nm +-50nm in between the projector source and the detector since we are most interested in the light intensity at the wavelength of ruthenium's peak absorption, approx 452nm.

II. Experimental Observations

A. On Excitability

1. How BZ responds to inhibitor concentration

2. Tongue bending & other tricks

B. Spatial & Stochastic Forcing

1.Preliminary results:

2.Labview instructions

(make random images using the makerandomimage.ni program in Labview.)