



## BDL-275-PPSX Batch Operating Sequence

Congratulations on joining the world of small scale Biodiesel Production; Take time to become intimately familiar with every aspect and feature of your new processor, find the location of each tagged valve and each one of the process buttons and switches. Each one of them plays an important part in the proper operation and function of the processor. If you feel comfortable with the location of all of the valves, buttons and switches it is time to proceed with making your first batch of Biodiesel.

- 1.** Check all isolation valves and make sure that they are in the open position and fill the Stage One Reaction vessel (preconditioning tank) with feedstock to the 1040 liter level
- 2.** Turn on the control power switch
- 3.** Press the preheat switch the unit will begin the preheat stage, if the oil is cold this stage could take several hours to complete when complete the preheat light will go out let the oil temperature fall to below 120 degrees F
- 4.** Drain off or pump off all accumulated water from the bottom of the vessel
- 5.** Transfer the oil into the Stage Two Reaction Vessel by pressing the Oil transfer switch the pump will shut off when the vessel is empty
- 6.** If three position selector switch is not already in the "A" or Re-heat position turn it to the "A" or Re-heat position now.
- 7.** Press the Re-heat button; This will enable the process to gently preheat the feedstock to 140 degrees F and maintain it at that temperature until you are ready to proceed to the next step
- 8.** Refill the stage one reaction vessel (preconditioning tank) with feedstock to the 1040 liter level
- 9.** Press the preheat switch the unit will begin the preheat stage, if the oil is cold this stage could take several hours to complete when complete the preheat light will go out; let the oil temperature fall to below 120 degrees F
- 10.** Drain off or pump off all accumulated water from the bottom of the vessel
- 11.** Once the feedstock in the stage two reaction vessel has reached temperature it is time to move on to the React phase of the process; Turn the three position selector switch to the "B" or react position
- 12.** Press and hold the Glycerin Drain button for 60 full seconds; this operation insures that you have true clean conditioned feedstock available in the suction manifold to draw a sample from
- 13.** Turn the three position selector switch back to the "A" or Pre-heat position and press the Preheat button to re-engage the Preheat cycle to maintain the feedstock temperature

14. Open the titration sample valve slowly and draw approx 400 milliliters of feedstock for a sample to titrate. Close valve
15. Titrate feedstock to determine the amount of KOH or NAOH to add to the methanol to generate the reaction process (see appendix A)
16. Open air valve and adjust regulator (if necessary) to establish air pump stroke rate of 2 strokes per second (note the maximum pumping rate is 2 strokes per second per pump manufacture) fill methyl/oxide mixer drum to the indicated 55 gallon mark
17. Close air valve
18. Weigh out KOH or NAOH as dictated by titration
19. Remove the vent plug from the methyl/oxide Mixer drum and using a wide mouth funnel pour in the prescribed amount of KOH or NAOH
20. Replace the vent plug; Note at all times when handling potentially dangerous chemicals such as methanol, KOH or NAOH it is recommended that safety precautions such as wearing protective gloves and a respirator be a matter of normal operation.
21. Turn the three position selector switch to the "B" or React position
22. Press the Mixer button; The Mixer runs for 7 minutes
23. After the Mixer times out Press the React button
24. open manual injection valve and press the Inject Button to start the injector pump and inject the methyl/oxide into the process pump suction stream
25. When all of the methyl/oxide has been injected into the feedstock the injector pump will automatically shut off, close the manual injection valve and (secure the bulk methanol drum and move it away from the processor)
26. The process pump will continue to run for approx 1 hour and will then time out; at this point the glycerin will begin to fall out of the raw biodiesel. The amount of time it will take for all of the glycerin to fall out of the raw biodiesel will vary from batch but should take at least 2 to 2 1/2 hours to complete
27. When you are reasonably sure all of the glycerin has fallen Press and hold the Glycerin button to pump out the glycerin out to the top of the stage one reaction vessel ( note you will have at a minimum 55 gallons to 60 gallons of glycerin to remove from the reactor) when you see the color phase change at the glycerin hose discharge release the Glycerin Drain button and (secure the glycerin container and move it away from the processor)
28. Press the pretreat button on the stage one vessel to allow the pretreat pump to operate it will time out in less than 15 minutes. at this point the glycerin will begin to fall out of the raw biodiesel. The amount of time it will take for all of the glycerin to fall out of the raw feedstock will vary from batch to batch but should take at least 1 to 2 hours to complete
29. Turn the three way selector switch to the "C" or Dry Wash position
30. Press the Dry Wash button; After about two minutes check the pressure gage on the methanol recovery heater the proper flow rate is 2 ½ lbs to 3 lbs although in the beginning of the Dry Wash cycle you may see pressures in excess of 4 lbs it will begin to fall as soon as negative pressure is developed inside the reactor. The pressure can be regulated by slowly moving bypass valve handle either toward the open or closed position in very small increments.

31. Open the water valve to establish cooling water flow through the methanol recovery condenser. The Dry Wash cycle will take approx 2 ½ hours more or less to complete do not get alarmed if the methanol recovery temperature does not climb to operating temperature immediately it will climb to operating temperature when there is available methanol to recover which may be in the second half of the batch
32. When you are reasonably sure all of the glycerin has fallen in the stage one reaction vessel Press and hold the Glycerin button to pump out the glycerin out to the Glycerin storage vessel ( note you will have at a minimum 55 gallons to 60 gallons of glycerin to remove from the reactor) when you see the color phase change at the glycerin hose discharge release the Glycerin Drain button and (secure the glycerin container and move it away from the processor)
33. After the Dry Wash cycle has ended it is advisable that you leave the finished biodiesel in the integrated storage tank until it has cooled to ambient temperature this will help to prevent the propagation of moisture in the fuel by exposing it to sudden cooling in the process of pumping and going into a cool vessel for final storage
34. Your processor is now ready to start your next batch of Biodiesel
35. After the finished Biodiesel has cooled turn on the Fuel Transfer switch to transfer the fuel to final storage

### Appendix A:

#### **Why We Titrate:**

The biodiesel reaction needs alkaline lye (NaOH) or KOH, as a catalyst (methanol and vegetable oil won't react to make biodiesel by themselves) Waste oil contains free fatty acids (FFA's), and the free fatty acids will mix with lye to make soap before the lye has a chance to participate in making biodiesel. We do a titration to find out how much free fatty acid is present and to find out how much to compensate for it by adding more lye so there's some left for the desired biodiesel reaction.

#### **How To Titrate:**

The titration performs the lye/free fatty acid reaction on a very small scale, and we use pH to measure it (somebody before us has previously figured out which pH change indicates that this reaction is complete, and it's at pH 8.5, the color change point of phenolphthalein indicator. Phenol red is close enough and is a hardware store item).

#### **How To Use The Information:**

The titration will give you a number (technically called acid value or acid number). We know that we can compensate for the fact that the free fatty acid will consume some of our lye, by adding a specific amount of lye to 'sacrifice' to the soap-making side reaction that the FFA's forces on us. The way this particular titration is written, every 1 ml titration result (ie the acid number) will tell us to add an extra 1 gram of lye for each liter of oil/ffa's you're using to make biodiesel to compensate for the side reaction caused by the FFA's.

## Titration for NaOH (Sodium Hydroxide)

### Step 1: Make Reference Tester Solution:

First, make a .1% NaOH in water solution- 1 gram of catalyst in 1 liter of distilled water. Try and be as accurate as possible with the measurement of the 1 gram of NaOH. Keep it sealed and it'll last for many titrations. You should be as accurate as possible when measuring the 1 gram of NaOH. You can improve your accuracy by measuring 3 grams of NaOH and adding it to three liters of distilled water, or some similar variation on that theme. Every 1 ml of this solution will now contain 1/1000 of a gram of NaOH- an amount too small to weigh. The water makes it possible to measure such tiny amounts of NaOH however. Keep this base solution in a bottle with a tightly closed lid, make new base solution every 30 days and make a new base solution each time you open a new bag of NaOH or KOH.

### Step 2- Perform a blank Titration:

Sometimes alcohol becomes slightly acidic with age, which would throw off your results. So we test it by performing a blank titration periodically. A blank titration looks just like a regular titration but without the oil. A blank titration neutralizes any acids that the isopropyl contained, so you're starting with a 'blank' slate and your real titration only reads the free fatty acids instead of the acids in the isopropyl. - Add 10 ml of isopropyl to a small 'beaker' - Add four drops of phenolphthalein or phenol red' - Swirl. It'll be some sort of yellow color' - Next, add NaOH/water drop by drop and keep swirling' - The moment it turns purple, stop- you've neutralized all the acids in the isopropyl. This is your starting point. You will now add oil to the mixture for the actual 'titration' step. If the isopropyl only needed 5 or 10 drops of NaOH/water solution to neutralize the acids, it's not very acidic. If it required a half milliliter or more of NaOH/water then that's more unusual (you should consider replacing your isopropyl alcohol). Perform a blank titration every time (it provides a more neutral starting point for the real titration) and the chance of batch failure will be greatly reduced.

### Step 3: Measuring Oil Sample:

Measure an exact 1 ml of oil with an oral syringe. Measure the oil with a different syringe than the isopropyl. The amount of oil is very crucial, but the isopropyl isn't. Add the 1 ml of oil to the 10 ml of alcohol that you have prepared above and stir. The liquid will be yellow after you've added the oil.

### Step 4: Add And Measure NaOH/Water Base Solution:

Now, add to this 'beaker', a small amount ( $\frac{1}{4}$  milliliter at a time) of the 0.1% NaOH solution drop by drop to the oil-alcohol-phenolphthalein mixture, stirring all the time. It might turn a bit cloudy, keep stirring. Keep on carefully adding the NaOH solution until the mixture starts to turn pink (magenta) and stays that way for 30 seconds of swirling. Don't mix up your oil and your lye/water syringes (clean them with isopropyl if you've made a mistake)

(Chopsticks make the best stirrers for titration.)

Take the number of milliliters of 0.1% NaOH solution you used and add **5** (the basic amount of NaOH needed for waste oil). This is the total number of grams of NaOH you'll need per liter of the oil you titrated.

## Titration for KOH (Potassium Hydroxide)

### Step 1: Make Reference Tester Solution:

First, make a .1% KOH in water solution- 1 gram of catalyst in 1 liter of distilled water. Try and be as accurate as possible with the measurement of the 1 gram of KOH. Keep it sealed and it'll last for many titrations. You should be as accurate as possible when measuring the 1 gram of KOH. You can improve your accuracy by measuring 3 grams of KOH and adding it to three liters of distilled water, or some similar variation on that theme. Every 1 ml of this solution will now contain 1/1000 of a gram of KOH- an amount too small to weigh. The water makes it possible to measure such tiny amounts of KOH however. Keep this base solution in a bottle with a tightly closed lid, make new base solution every 30 days and make a new base solution each time you open a new bag of NaOH or KOH.

### Step 2- Perform a blank Titration:

Sometimes alcohol becomes slightly acidic with age, which would throw off your results. So we test it by performing a blank titration periodically. A blank titration looks just like a regular titration but without the oil. A blank titration neutralizes any acids that the isopropyl contained, so you're starting with a 'blank' slate and your real titration only reads the free fatty acids instead of the acids in the isopropyl. - Add 10 ml of isopropyl to a small 'beaker' - Add four drops of phenolphthalein or phenol red' - Swirl. It'll be some sort of yellow color' - Next, add KOH/water drop by drop and keep swirling' - The moment it turns purple, stop- you've neutralized all the acids in the isopropyl. This is your starting point. You will now add oil to the mixture for the actual 'titration' step. If the isopropyl only needed 5 or 10 drops of KOH/water solution to neutralize the acids, it's not very acidic. If it required a half milliliter or more of KOH/water then that's more unusual (you should consider replacing your isopropyl alcohol). Perform a blank titration every time (it provides a more neutral starting point for the real titration) and the chance of batch failure will be greatly reduced.

### Step 3: Measuring Oil Sample:

Measure an exact 1 ml of oil with an oral syringe. Measure the oil with a different syringe than the isopropyl. The amount of oil is very crucial, but the isopropyl isn't. Add the 1 ml of oil to the 10 ml of alcohol that you have prepared above and stir. The liquid will be yellow after you've added the oil.

### Step 4: Add And Measure KOH/Water Base Solution:

Now, add to this 'beaker', a small amount ( $\frac{1}{4}$  milliliter at a time) of the 0.1% KOH solution drop by drop to the oil-alcohol-phenolphthalein mixture, stirring all the time. It might turn a bit cloudy, keep stirring. Keep on carefully adding the KOH solution until the mixture starts to turn pink (magenta) and stays that way for 30 seconds of swirling. Don't mix up your oil and your lye/water syringes (clean them with isopropyl if you've made a mistake)

(Chopsticks make the best stirrers for titration.)

Take the number of milliliters' of 0.1% KOH solution you used and add **7 - 8** (the basic amount of KOH needed for waste oil). This is the total number of grams of KOH you'll need per liter of the oil you titrated.

To confuse matters further, KOH comes in a variety of purities. You want an 85% or higher. However for beginners we recommend just buying the 99% pure. Later on you

can adjust your KOH levels to compensate for impurities (ie if you've got 90% pure KOH use 10% more of it and so on). Do not adjust the results of the titration! the titration will automatically reflect the impurity level for you.

275 US gallons = 1040.93 liters