Atlas Sodium Automated Batch Synthesis System (Syrris)
June 2013
Introduction to the system

- The Atlas Sodium system consists of an Atlas base equipped with a 400°C hotplate, a stacking dry bath system for round-bottomed flasks of 50mL to 1L in size, an overhead stirrer with speeds up to ----rpm, a chemically resistant temperature probe, an optional pH probe, and a syringe pump with 2 glass syringes (5mL and 0.5mL) for automated addition and/or removal of solution.

- The system is computer controlled by the Atlas software which allows for real-time data logging of multiple variables every second and creation of detailed recipes for conducting synthesis or monitoring reaction conditions.

- Some feature of the system are controlled manually using the red buttons and selection wheel on the syringe pump and atlas base.
Start Up

• Turn on power for the Syringe Pump (back, upper left corner)
• The screen on the pump will display the message as seen below → Press the large red button to confirm
Start Up, cont.

• Turn on the power for the Atlas base system (lower left side)

• If you will be running a reaction at an elevated temperature we recommend manually setting the hotplate temperature at system start-up to reduce temperature ramp-up time when running a recipe.

• Use the selection wheel to place the → ← arrows around the temperature setting, then press the enter button, then turn the selection wheel until the desired temperature is displayed and press the enter button.
Defining the Apparatus

- Open the Atlas software on the computer to the left of the fume hood
- Login using your username and password
- In the apparatus tab on the left side of the screen specify which system items you will be using for the reaction by dragging and dropping them onto the diagram on the right
  - Set the device properties for each item
  - The Base Node Port for the temperature and pH probes should be set as **Syringe Pump 1 Port 1**
  - Items that have been selected for use are displayed as icons on the bottom left side of the screen
Creating your Recipe

• In the recipe tab drag and drop a step type into the recipe screen on the right → for each step you will need to input your operating parameters
• Repeat for all desired reaction steps (for examples see the developed basic protocols)
  • Typical starting steps including setting a stirrer speed and a target temperature
  • Temperature can be controlled by monitoring the hotplate temperature or by monitoring the reaction temperature with the temperature probe *Note that reaction temperatures tend to be ~40°C cooler than the hotplate temperature*
  • Custom end points and limits may be defined using basic logic available in the step properties window
Creating your Recipe, cont.

• The syringe pump can be used in four different modes: Fill, Empty, Pump, and Continuous Pump.
• Fill and Empty pump modes are useful for flushing out the tubing before pumping a solution into the reaction vessel.
• Pump mode is useful for addition of a specific volume of a solution.

• The pump has two channels and each channel has 6 ports (A, B, C, D, E, F).
  • Port E on both pump channels is reserved for filling of a cleaning solution (water or 1% nitric acid).
  • Port D on both pump channels is reserved for emptying waste (for flushing and cleaning tubes).
  • Ports A, B, C & D on both pump channels should be used in pairs (one port for filling, one for emptying) for addition of reaction solutions.
Creating your Recipe, cont.

• Limits and end conditions can be added to the syringe pump properties to ensure that the rate of addition of a solution is sufficiently slow to reduce temperature fluctuations

• The syringe pump can also be operated in pH control mode for titrations

• Multiple simultaneous pump steps may be entered in a recipe by dragging and dropping a new step to the side of a current step instead of dropping it above or below the current step.

• Save your recipe
Preparing to Run your Reaction

• Raise the Stirrer assembly so that you can place your reaction vessel underneath.

• Raise the flask clamp assembly high enough that you can press the neck of the flask into the spring clamp → tighten the spring clamp → lower the spring clamp assembly with your reaction vessel into the dry bath → tighten the screw clamp to secure

• Lower the stirrer assembly until the stirrer is near the bottom of your reaction vessel → either tighten the screw clamp to secure OR allow to rest on top of the flask clamp assembly

• Place the temperature probe and/or pH probe in your reaction vessel and secure them in a location where they will not be hit by the stirrer rod.
Preparing to Run your Reaction, cont.

• If using the syringe pump first flush the input lines with the appropriate solution
• Place your input line in your solution vial and your output line in a waste collection vessel
• Use the selection wheel to select Pump 1 (left) or Pump 2 (right) → select “Flow at rate” → choose an appropriate flow rate for the syringe volume → select the input and output port (default is A in and B out) → press the Enter button
• Let run until no bubbles are seen in the input and output lines then go to Pump → stop
• Repeat for all solution input/output sets (the last solution flushed should be the first one you want to dispense. A maximum of 2 solutions can be dispensed at one time with a total of 6 different solutions that may be dispensed during a reaction.
• Place your input lines in your reaction vessel and secure them.
Running your Reaction

- In the Atlas software go to the Run Experiment tab and click Start.
- You can monitor reaction parameters in real-time by dragging and dropping a parameter from the list on the left into the graphics slot below the system diagram and recipe flow chart.
- You can enter comments in real time (time stamped) to add to your data file in the box in the upper right.
Collecting your Data

- To get a complete text file of your logged data go to C:\Program Files\Atlas1.4\Logs and open the most recent log file (every reaction log run is automatically saved here)
Shutdown

- Log out of the Atlas software
- Remove your reaction vessel from the Atlas base, if your vessel is hot raise the spring clamp assembly out of the dry bath and allow to cool before removing
- Place the designated cleaning vessel filled with water in the atlas base and submerge the stirrer and any used probes and syringe pump input/output lines in the water
- On the Atlas base select the stirrer speed and set to 500rpm (leave the hotplate off)
- If the syringe pump was used use the pump manual controls to turn on the pump flow.
- Allow to wash for 10 minutes
- Turn off stirrer and pump, remove the cleaning vessel, dump the wash water, rinse the vessel, then refill with clean water and place back on the Atlas system and repeat cleaning step for at least 5 minutes
- When done with second cleaning switch off the power to the syringe pump and Atlas base, remove the cleaning vessel and dump the water and rinse the vessel
- If the stirrer, probe, or syringe pump lines are still visibly dirty repeat cleaning with a 1% Nitric Acid Solution for 30 minutes followed by an additional 10 min cleaning in water
- Power off the Atlas Base and Syringe Pump