

START Class Lab
March 13, 2013

Operating an Automated Synthesizer – ELIXYS

Purpose:

This lab is designed to help radiation workers be familiarized with the planning, preparation, and operation of the ELIXYS fully-automated radiosynthesizer by designing, programming, and running the ELIXYS using information typically found in literature.

Procedure:

Using the information below, each group will design a synthesis protocol that can be programmed into the ELIXYS software. First, on paper, create a synthesis “workflow” using the unit operations and their respective parameters that have been summarized on the back of this page. You can use the space provided on the back of this page as a template. Then, you will use the ELIXYS software to program your synthesis protocol into a “sequence”. When you have completed the sequence, you will prepare the necessary reagent vials by filling them and crimping them closed. Once you are ready to begin synthesis, you will load the necessary components (e.g. reagent vials, reactor vials, etc.) into the ELIXYS and run the sequence.

Synthesis Protocol:

Materials:

Reagents A, B, C and Solvent were prepared by Mark Lazari and used as received. Alumina-N solid phase extraction (SPE) cartridges were purchased from Waters and conditioned using 10mL of 18MΩ water. The ELIXYS radiosynthesizer, reactor vial, collection vial, stir bar, cassette, and the reagent vial preparation kit (amber 3mL reactor vials, rubber septa, 13mm crimp top caps, and crimper) were provided by the van Dam lab.

Synthesis:

Reagent vials were loaded as follows: 1.0mL of reagent A (vial 1), 0.8mL of reagent B (vial 2), 0.8mL of reagent C (vial 3), 2mL of solvent (vial 4). Reactor vial with stir bar was installed in reactor 1 and contents of vial 1 was added to the reactor vial. Reactor contents were evaporated to dryness at 110°C. Contents of vial 2 was then added and the solution reacted at 130°C for 5min. The reaction solution was partially evaporated at 100°C and then the contents of vial 3 was added. The new reaction solution was reacted at 80°C for 5min in a separate sealed reaction position. The resulting crude product was purified by passing through an Alumina-N SPE cartridge into a clean collection vessel. The contents of vial 4 was added and used to rinse the SPE cartridge to flush out remaining purified product to the collection vessel.

Table of pertinent unit operations and their necessary parameters

| Unit operation | Parameter | Description |
|---|---|--|
| ADD Adds a reagent to a reaction vessel | Reagent Reactor Delivery position | Reagent to add Reactor to which the reagent will be added Choice of two separate delivery positions |
| EVAPORATE Evaporates the contents of a reaction vessel | Reactor Evaporation temp Evaporation pressure Final temp Duration Stir speed | Reactor to evaporate Temperature to heat to during evaporation (Celsius) Inert gas pressure to use when evaporating (PSI) Temperature to cool to after evaporation (Celsius) Time to evaporate after reaching temp (seconds) Rate of stirring during evaporation |
| TRANSFER Transfers the contents of one reactor to the next (optionally through a purification cartridge) | Source reactor Target reactor Mode | Reactor to transfer from Reactor to transfer to "Trap" to send to waste or "Elute" to send to the next reactor |
| REACT Seals the reactor and heats | Reactor Reaction temp Reaction position Duration Final temp Stir speed | Reactor to heat Temperature to heat to during reaction (Celsius) Choice of two separate reaction positions to avoid cross-contamination if multiple sealed reactions are performed in the same vessel Time to react after reaching temperature (seconds) Temperature to cool to after reaction (Celsius) Rate of stirring during reaction |
| PROMPT Pauses the sequence run and prompt the user | Message | Message to display when prompting the user |
| INSTALL Moves the reactor to the install position for reaction vessel removal and/or installation, and prompts the user | Reactor Message | Reactor to move to the install position Message to display when prompting the user |

Workflow:

| | Unit Operation | Parameter Information | | Unit Operation | Parameter Information |
|---|----------------|--|----|----------------|-----------------------|
| 1 | Initialize | (You must use this as your first unit operation) | 10 | | |
| 2 | Add | Reagent A, 15s, 3 PSI | 11 | | |
| 3 | | | 12 | | |
| 4 | | | 13 | | |
| 5 | | | 14 | | |
| 6 | | | 15 | | |
| 7 | | | 16 | | |
| 8 | | | 17 | | |
| 9 | | | 18 | | |