Oil-Free, Energy-Efficient Heating Blocks to Streamline Synthetic Chemistry

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Description: Many chemical reactions require heating, sometimes continuously for several days or at temperatures over 100 ºC. Typically, silicone oil baths are used to heat these reactions as silicone can withstand high heats without risk of polymerization. However, the production and use of silicone oil presents an environmental hazard and is a significant source of energy usage in the lab. Aluminum heating blocks are a more energy-efficient alternative to oil baths; aluminum has a higher thermal conductivity and specific heat capacity than silicone oil and thus less energy is required to maintain a desired temperature. Our project aims to move towards eliminating oil heating baths from our lab by purchasing sets of aluminum heating blocks for each workstation of our lab.

Objective: Use aluminum heating blocks to replace the energy-intensive silicone oil baths used for heating chemical reactions.

Results: To encourage our lab to move away from conventional oil heating baths, we purchased heating blocks that fit (1) a 100ml round bottom flask or (2) 4 x 20 ml vials for each chemical hood in our lab, in addition to a few additional aluminum heating blocks for larger size (250+ ml) flasks and smaller (>10ml) vials for high-throughput screening. Overall, we feel that this was highly successful as nearly everyone routinely uses these heating blocks over the silicone oil blocks, leading to lower energy consumption in our lab. We discovered a few key additional takeaways from this project:

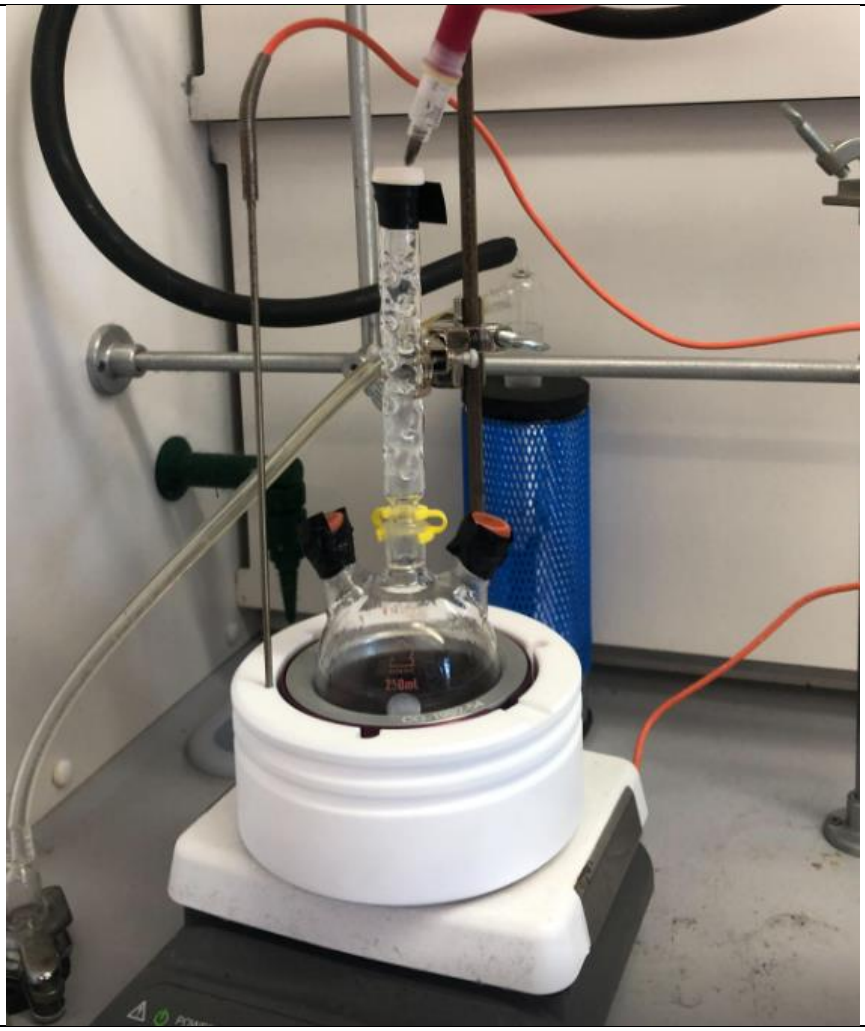
(1) **Heating blocks simplify reaction setup.** Oil baths require careful placement of temperature probes and thermometers into the oil, which can complicate the setup of reactions especially when larger and more complicated glassware is needed. Additionally**, larger more cumbersome oil baths are needed for larger size reactions that require disproportionate amounts of oil to fully submerge the flask.** In contrast flasks or **vials are easily inserted into the pre-fit aluminum heating blocks, and the larger size heating blocks only cover the surface of the flaks**. See example picture (1)

(2) **Heating blocks significantly increase reaction throughput.** Because many of the heating blocks that we purchased can accommodate multiple reaction vials at a time, we can set up high-throughput experiments to optimize or explore scope of chemical reactions more quickly. **This dramatically reduces the number of heating pads needed and energy used to set up high-throughput**

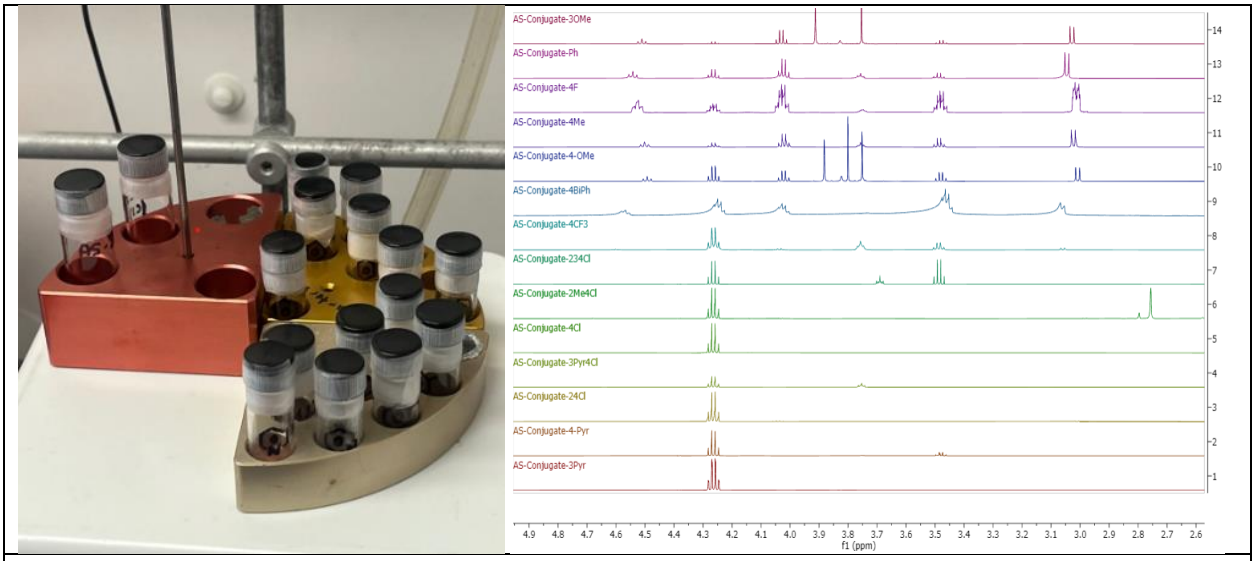
experiments, as oil baths can (safely) hold a maximum of 2-3 reactions at a time. By miniaturizing these reactions, **we can also substantially reduce the scale of our reactions which reduces the cost of expensive chemicals including palladium, solvents, etc**. For an example of high-throughput experimentation (>20mg scale each), see example picture 2.

(3) Heating blocks are safer than traditional oil baths. When using our aluminum heating blocks, the temperature probe is securely inserted into the base of the block (see related media). In contrast the temperature probe in oil baths are loose, and dangerous over-heating events can occur if this temperature probe is accidentally moved or bumped when working in the hood. **By using these aluminum heating blocks with secure temperature probe inlets, we feel that we are at significantly less risk of these overheating events.**

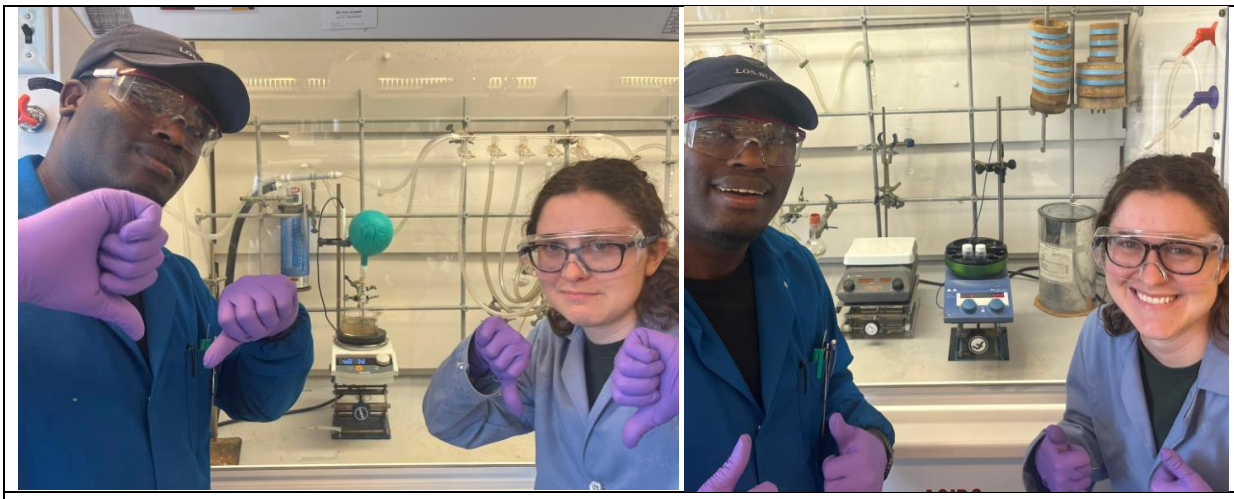
Related Media



(1) Example reaction setup: Decagram-scale reflux (120dC); no oil needed!



(2) High-Throughput Experimentation: 16 reactions at once saves significant time, energy, and space. Example 1H-NMR data generated in a single day from high-throughput screening of a Pd-catalyzed conjugate addition



(3) Before and after replacing oil baths with energy-reducing, safe, and high throughput heating blocks