Experiment 29

MICROWAVE REACTIONS: A RESEARCH PROJECT



Drs. Philip Myhre and the late Nien-Chu Yang and George Hammond were my (this author's)

Discussion

The experiments you have been performing in this organic laboratory course have been designed to give you experience in the field of organic chemistry. Because of the potential risks and the cost of chemicals, the previous experiments gave you detailed directions that were carefully designed to minimize risk. However, every experiment was selected because it had a challenge associated with it. Now it is time to give you more of the responsibility. With the responsibility, there is an increased possibility that you will feel the excitement that results from exploring the frontiers of human knowledge. Running an experiment that to your knowledge has never been performed before should provide a special sense of satisfaction to you.

The responsibilities associated with probing the unknown must be taken seriously. You must read the pertinent literature so that you can maximize the chances of success and minimize the risk. You must discuss your plans with your instructor to determine if your research proposal is suitable and safe for your laboratory environment. To make this experiment manageable in your laboratory, the choice of reactions will be limited to those that might work in a microwave oven. Microwave assisted organic reactions are a fairly recent addition to the repertoire of techniques available to organic chemists and therefore still a rather fertile field for research. Microwave reactions that work generally take only a few minutes and therefore can be run during the time restrictions imposed by a college laboratory course. An example of a microwave assisted reaction can be found in *Experiment 24*. The very recent article by Jensen, et. al. below also contains some suggestions. For more instructions, see the *Prelaboratory Preparation* section of this experiment and use the references below.

E29-2

References

<u>textbook</u>

Leadbeater, N. E.; McGowan, C.B. Laboratory Experiments Using Microwave Heating, CRC Press, 2013.

reactions

Reilly, M. K.; King, R. P.; Wagner, J.; King, S. M. J. Chem. Educ., 2014, 91, 1706 - 1709.

- Baar, M. R.; Gammerdinger, W.; Leap, J.; Morales, E.; Shikora, J. J. Chem. Educ., 2014, 91, 1720 1724.
- Russell, C. B.; Mason, J. D.; Bean, T. G.; Murphree, S. S. J. Chem. Educ., 2014, 91, 511 517.
- Elder, John W.; Holtz, Kathleen M. J. Chem. Educ., 1996, 73. A104.

Bari, S. S.; Bose, A. K.; Chaudhary, A. G.; Manhas, M. S.; Raju, V. X.; Robb, E. W. J. Chem. Educ., 1992, 69, 938 - 939.

Freeman, R. G.; McCurdy, D. L. J. Chem. Educ., 1998, 75, 1033 - 1034.

Banik, B. K.; Barakat, K. J.; Wagle, D. R.; Manhas, M. S.; Bose, A. K. J. Org. Chem., 1999, 64, 5746 - 5753.

Mirafzal, G. A.; Summer, J. M. J. Educ. Chem., 2000, 77, 356 - 357.

Trehan, I. R.; Brar, J. S.; Arora, A. K.; Kad, G. L. J. Chem. Educ., 1997, 74, 324.

Elder, J. W. J. Chem. Educ., 1994, 71, A142.

Murphree, S. S.; Kappe, C. O. J. Chem. Educ., 2009, 86, 227-229.

http://www.mdpi.org/ecsoc-5/e0000/e0000.htm (accessed 04/30/10)

Jensen, J.; Grundy, S. C.; Bretz, L.; Hartley, C. S. J. Chem. Educ., 2011, 88, 1133.

Damkaci, F.; Dallas, M.; Wagner, M. J. Chem. Educ., 2013, 90, 390 - 392.

Baar, M. R. J. Educ. Chem., 2018, 95, 1235 - 1237.

the microwave oven

Villemin, D.; Thibault-Starzyk, F., J. Chem. Educ., 1991, 68, 346.

Cresswell, Sarah L.; Haswell, Stephen J., J. Chem. Educ., 2001, 78, 900 - 904.

reviews

Cundy, C. S. Collect. Czech. Chem. Commun., 1998, 63, 1699 - 1723.

Caddick, S. Tetrahedron, 1995, 51, 10403 - 10432.

Whittaker, G. http://homepages.ed.ac.uk/ah05/ch1/c1org.htm

Whittaker, G. http://homepages.ed.ac.uk/ah05/ch1a.html

Loupy, A. editor, Microwaves in Organic Synthesis, Wiley, 2002.

Bogda., D. Microwave Assisted Organic Synthesis, 25, Elsevier, 2005.

Tierney, J.P.; Lidström, P. Microwave Assisted Organic Synthesis, Blackwell, 2005.

Kappe, C. O. Angew. Chem. Int. Ed., 2004, 43, 6250-6284.

Romanova, N. N.; Gravis, A. G.; Zyk, N. V. Russ. Chem. Rev., 2005, 74, 969-1013.

miscellaneous

http://www.organic-chemistry.org/topics/microwave-synthesis.shtm

http://www.milestonesci.com/synth-fund.php

http://www.tan-delta.com/

http://en.wikipedia.org/wiki/Microwave_chemistry

Obermayer, D.; Gutman, B.; Kappe, C. O. Angewandte Chem. Int. Ed., 2009, 48, 8321-8324.

Koch, A. S.: Chimento, C. A.; Berg, A. N.; Mughal, F. D.; Spencer, J-P.; Hovland, D. E.; Mbadugha, B.;

microwave assisted extraction - Hovland, A. K.; Eller, L. R. J. Chem. Educ., 2015, 92, 170-174.

Prelaboratory Preparation - Experiment 29

First, be sure to list all the goals of the experiment. Perform a literature search for microwave assisted reactions and determine the kinds of reactions that have been demonstrated to work in a microwave oven. Solvent selection is very important for microwave assisted reactions and needs to be seriously considered. You should check on the cost, safety and availability (does your stockroom have the chemicals you need) of the chemicals. Try to find a reaction that interests you (is the product related to a pharmaceutical product or a chemical of environmental interest) and has a significant chance of success. Write up a research proposal for your reaction and submit the plan to your instructor several days before your scheduled laboratory meeting. If approved, try the reaction. If you are successful in isolating product, try the reaction again this time varying the power setting or microwave time and try to adjust the conditions to maximize the yield.

Procedure and Observations

Be sure to include a detailed procedure in this report. Report all relevant observations including the type of microwave oven, power settings and times.

Conclusions

This section should include the following:

- 1. Were the goals of the experiment achieved? Explain your answer.
- 2. What was the identity of your product and did it agree with your prediction? Explain your answer.
- 3. Compare microwave techniques to conventional heating techniques. What are the advantages and limitations of microwave techniques?
- 4. How could the percent yield and recoveries have been improved?
- 5. Write up the procedure as an experiment for your organic chemistry course with an introduction, discussion, procedure and appropriate questions.