Synthesis of Methyl Orange

Anna Hughes
Experiment Dates: 4/15/24 and 2/22/24

General Info

Goal:

- Synthesize methyl orange dye and test on different fiber types
- Test mordants to see color changes and test dye adherence to different fiber types

Uses of Methyl Orange:

- Dye
- Acid/Base indicator[1]
- Stain for microscopy[2]

General Info

How formed synthesis?

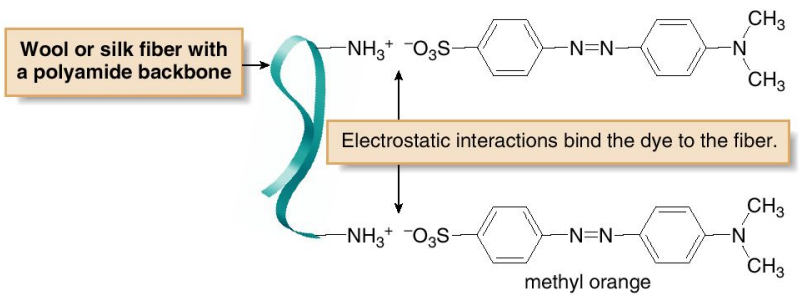
- Textbook Examples
- Lab Notebook Examples
- No current journal examples
  - Trying to eliminate methyl orange from waterways
  - Many papers on how to destroy via photocatalytic degradation

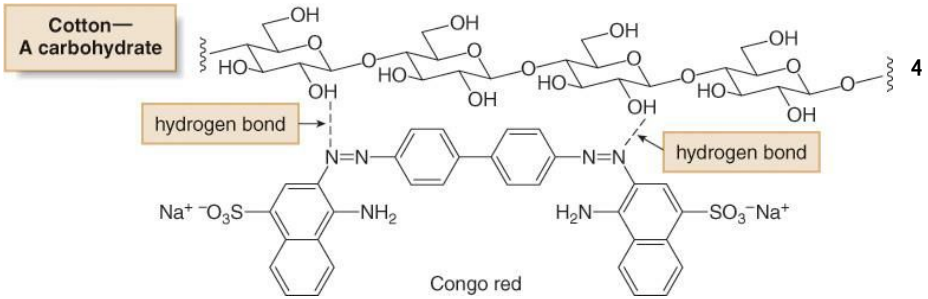
How relate to CHEM 2255?

- Azo coupling reaction - studied in lecture
- EAS reaction variation
  - Easy to accomplish in time period and skill level
  - Common industrial reaction

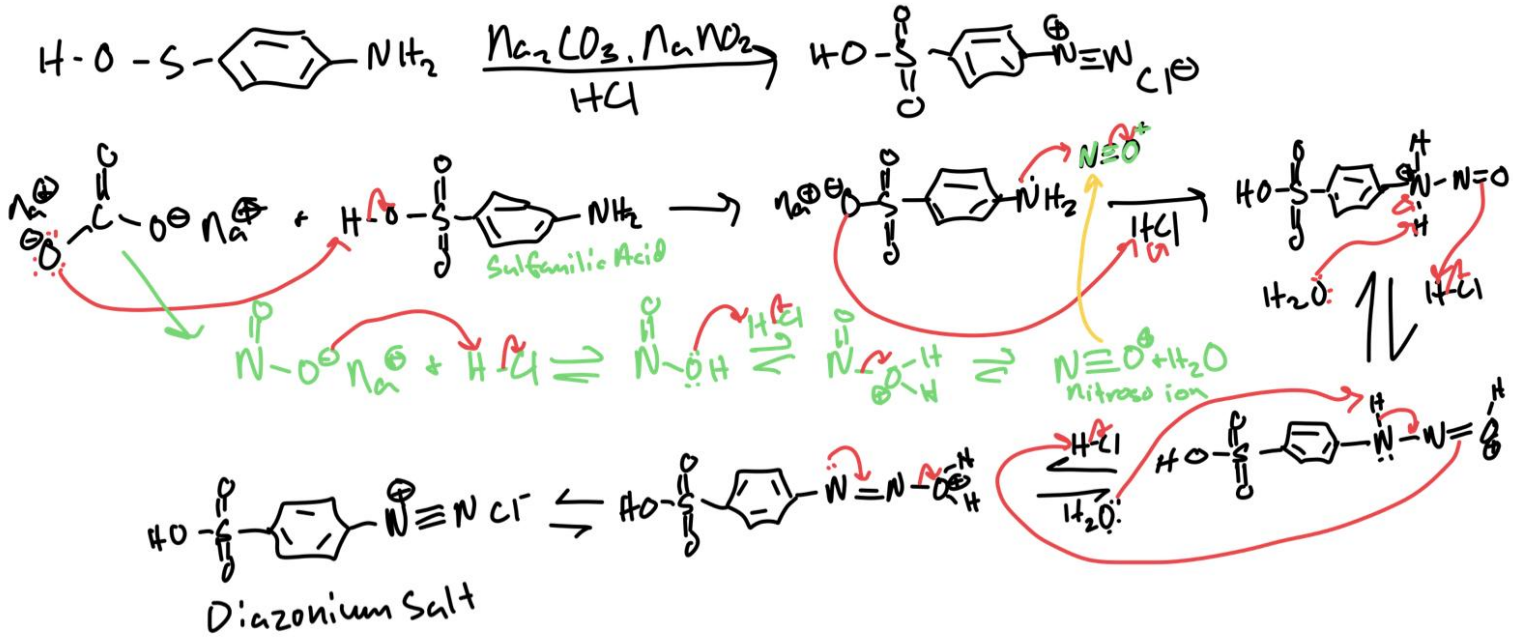
Background of Reaction

- Common azo dye (N=N)
- Used in industry today[2]
- Good experiment for class - visual changes[3]
- Direct dye - no mordant to prep[1]
    - Attaches directly to fiber

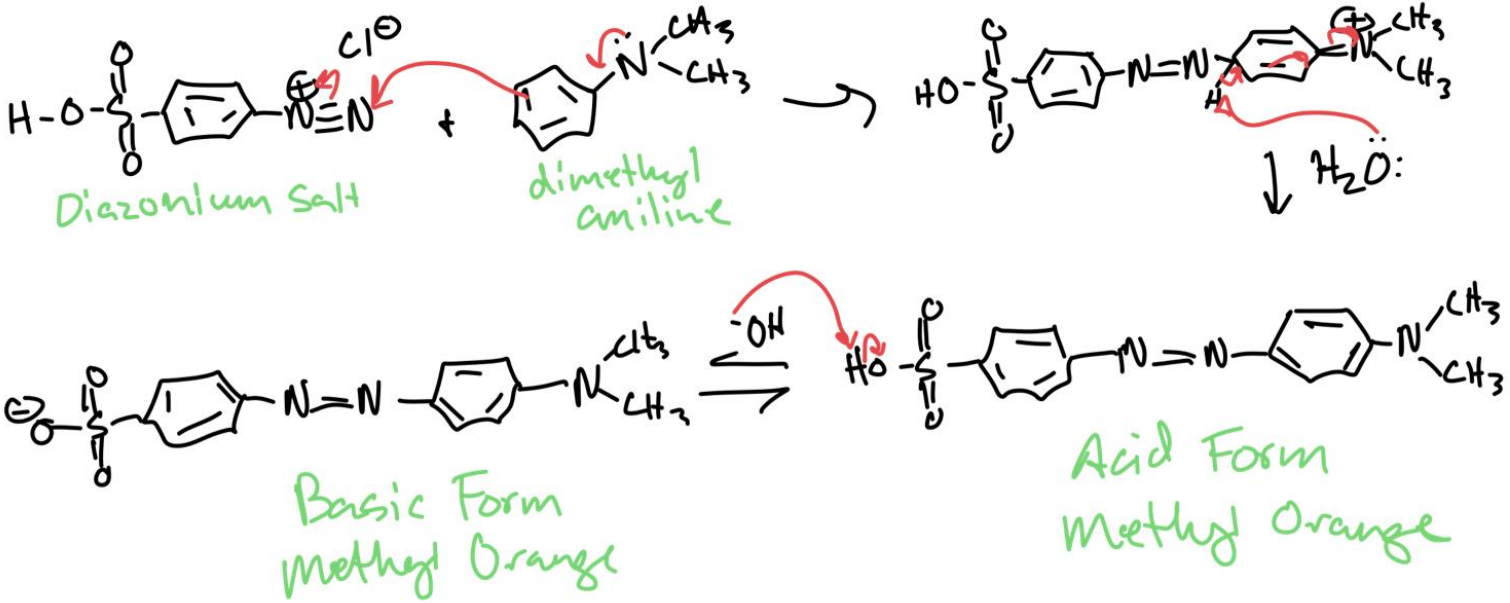


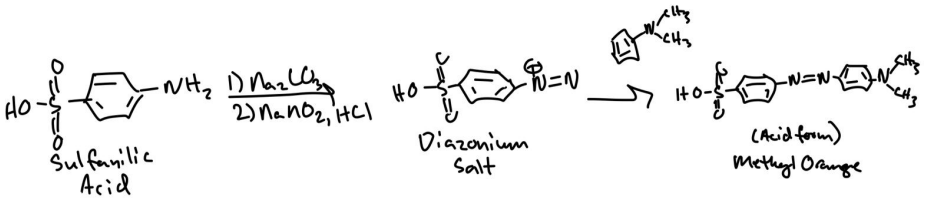


Synthesis of Methyl Orange Mechanism - Diazonium Salt



Synthesis of Methyl Orange Mechanism - EAS





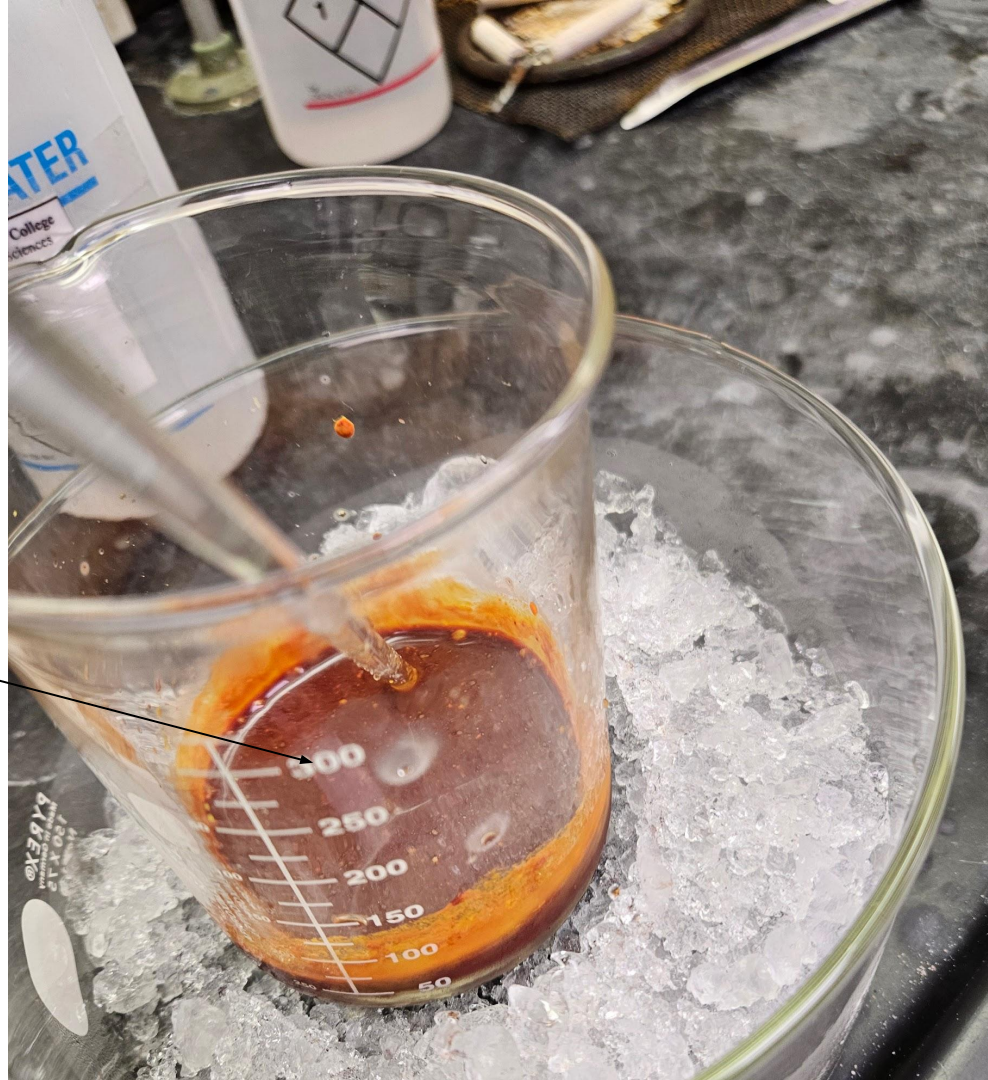
Experiment - Day 1

Synthesis of Diazonium Salt

1. Sulfanilic acid (2.461g) and sodium carbonate (0.66g) mixed and lightly heated to dissolve in 25mL water - bubbles evolved
2. Return to rt and add sodium nitrite (0.948g) - color change to orangey yellow
3. Pour over ice and add HCl (2.5 mL) while stirring. Keep in ice bath. Turned a little orange and cloudy with HCl.
4. This is diazonium salt.

Experiment Day 1

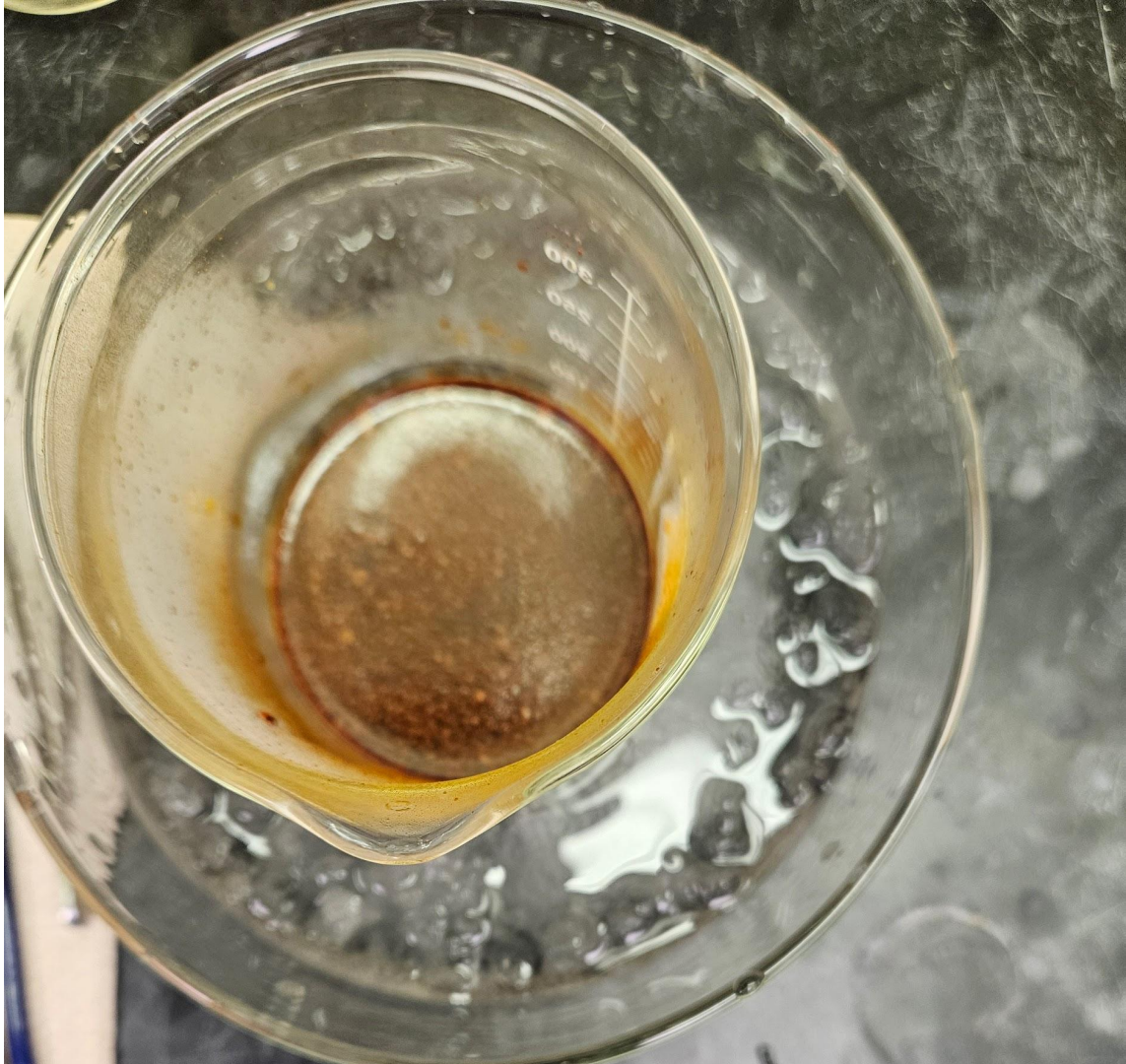
1. Mix dimethylaniline (1.6mL) and glacial acetic acid (1.25 mL) in a small flask.
2. Add this to diazonium salt mixture from last part.
3. KEEP STIRRING!
4. Acid form of dye will form - also make stiff paste.
5. Add sodium hydroxide (17.5 mL, 3M) to produce salt form.
6. Heat to boiling point and then cool UNDISTURBED.
7. Use saturated salt water to rinse when pulling solid.
8. Recrystallization failed first time - lost almost all product!!!!



Acidic Form of Methyl Orange before recrystallization

After Adding sodium Hydroxide

After Heating



After Ice Bath

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After Filtration
End of Day 1
Crude wt: 3.192g

Methyl Orange
Pure: 1.929g
94.65% yield

Day 2

1. Recrystallize trial and error - worked with combo of 2:1 ethanol and water
2. Dye made and test as indicator.
3. No time to mordant - skipped entire process - plus some nasty chemicals
4. Make dye bath containing

- 100 mg of Methyl Orange
- 1mL 3M sodium sulfate solution
- 30 mL water
- 10 drops 3M sulfuric acid

5. Heat till near boiling point and immerse fabric for 5 min.

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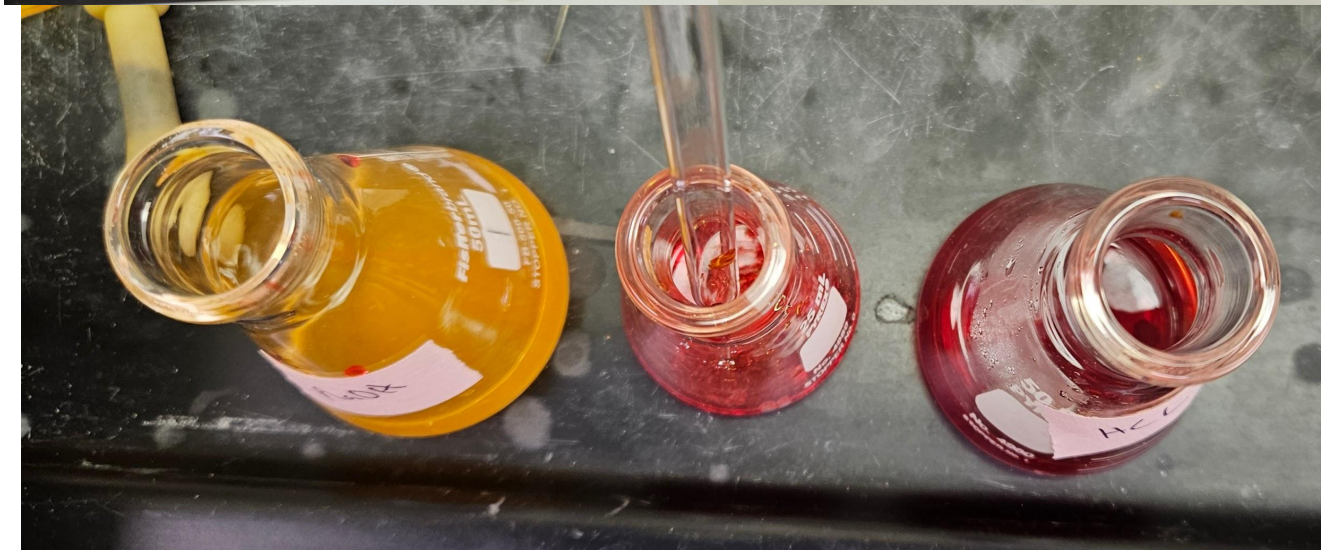
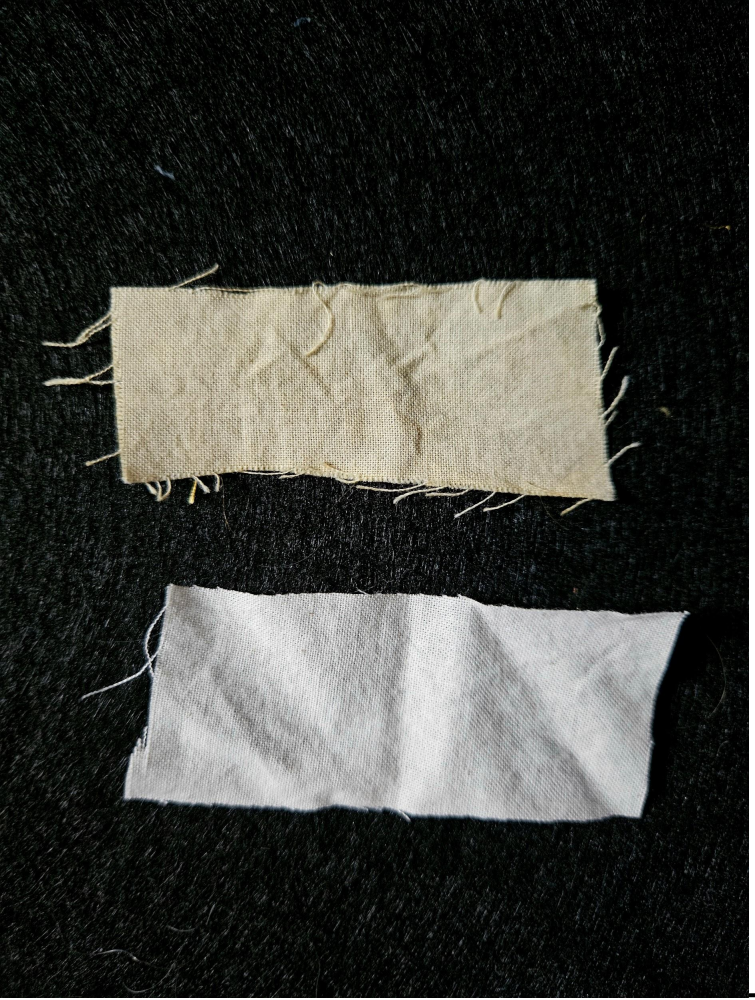
Testing as Indicator



Silk before and after

Protein Fibers

Wool before and after



Muslin - natural

Cellulose Fibers

Cotton/Poly blend

Cotton

Silk

Polyester

Acrylic Felt

Synthetic Fibers

Polyester/Cotton/Silk

Conclusions

- Yield much better than expected - water and ethanol mix worked well to recrystallize
- To further the experiment, having the ability to test mordants would be nice
  - try safer ones
- I would have rinsed the filter paper into the beaker if I had thought of it
  - better yield for crude product

Conclusions cont'd

- Fun lab to run in 2255, but very easy to get messy fast
  - Issues of supplying fabric samples and testing for fiber content (burn tests)
- Easy EAS lab with fewer side products unlike earlier nitration lab
  - It is a salt, so no mp verification
- Also cannot test with TLC to make sure diazonium salt is completely reacted
  - explosive when dry
- Synthesize different kinds of dyes - direct, vat, substantive, developed, disperse, fiber reactive
- Talk about hydrazone effects - new way to refer to azo pigments - different mechanism[5]
- Study environmental effects of industrial dyeing[2]
  - How to destroy synthetic dyes

References

1. Williamson, K. L.; Dyes and Dyeing. In *Macroscale and Microscale Organic Experiments*. D. C. Heath and Company: United States of America, **1989**. 604-621.
2. He,Y., Chen, M., Wang, J., Zhao, G., Han, S., Xu, Y., Chen, Y., Wang, C., Wang, J.A Comprehensive Chemistry Experiment for Undergraduates to Investigate the Photodegradation of Organic Dyes by ZnO/GO Nanocomposite. *J. Chem. Educ.* **2023.** *100 (9)*, 3556-3563.
3. Gung, B. W., Taylor, R. T. Parallel Combinatorial Synthesis of Azo Dyes: A Combinatorial Experiment Suitable for Undergraduate Laboratories. *J. Chem. Educ.* **2004** *81 (11)*, 1630-1632.
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5. Hunger, K.; Schmidt, M. U.; Heber, T.; Reisinger, F.; Wannemacher, S.; Hydrazone Pigments (Formerly Called Azo Pigments). In I*ndustrial Organic Pigments*, 4th Edition;Wiley-VCH Verlag GmbH & Co. KGaA.: Weinheim, Germany. **2018**.