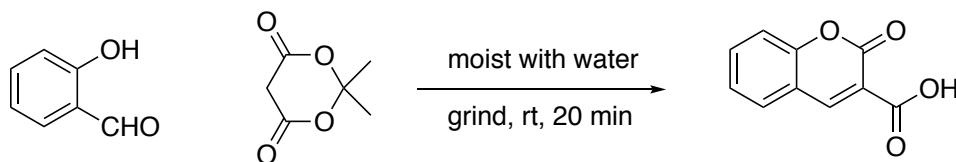


Mechanochemical synthesis of coumarin-3-carboxylic acid using a modified grind technique

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In this method, a unique method of reaction is demonstrated – mechanochemical reaction, sometimes called grindstone chemistry – which is promoted by thoroughly grinding the reactants with one another rather than normal methods. Grindstone chemistry eliminates the use of solvent which provides a greener reaction, since less waste is usually produced.

Specifically, we will follow the method published for the synthesis of 3-carboxycoumarin originally reported by Kumar, Kumar, and Makrandi¹ with the purpose of executing an experiment that exhibits the principles of green chemistry and can be easily performed in organic chemistry laboratories.



Prelab questions

1. Which of the reactants is called Meldrum's acid?
2. Write out the balanced reaction.
3. Calculate the atom economy of this method.
4. Convert the reactant mmols to volume (if liquid) or mass (if solid).
5. What is the theoretical yield of product in mass.
6. Download the article that this lab was based upon using the link.² In our example lab, $R_1 = R_2 = R_3 = H$. What is the literature melting point that we should expect?
7. What green principles are demonstrated in this method?

Procedure (CAUTION: product is toxic if swallowed; avoid contact!)

To a small mortar is added salicylaldehyde (3 mmol), Meldrum's acid (3 mmol) and 8 drops of water. The substances are thoroughly ground in mortar & pestle for 20 minutes before being allowed to sit for 40 minutes.

Next, a small amount (3-5 mL) of ethanol can be helpful to move the crystals from the mortar to the a test tube or vial. The product is recrystallized with the ethanol. After drying, the mass, melting point and IR spectrum are recorded. Calculate percentage yield and E factor.

References

1. Kumar, D.; Kumar, S.; Makrandi, J. K., Aqueous-mediated green synthesis of 3-carboxycoumarins using grinding technique. *Green Chem. Lett. Rev.* 2015, 8, 21–25.
<http://www.tandfonline.com/doi/pdf/10.1080/17518253.2015.1058975>