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Preparation of methyl salicylate, by DARKBLADE48

Here's a simple synthesis of methyl salicylate (oil of wintergreen), for those that just started (organic) chemistry (though of course, even if you are a bit more experienced, nothing beats a minty smelling flask/test tube).

Necessary reagents

Methanol, CH_3OH
Salicylic acid, $\text{OHC}_6\text{H}_4\text{COOH}$
Concentrated sulfuric Acid, H_2SO_4

Outline of procedure

The procedure is a Fischer esterification and is quite simple, and can be followed below. Note that I did this on a microscale, but if you have the right apparatus, this can easily be scaled up. You could also get more complicated by using a Dean-Stark trap for esterification, etc.

- 1) Dissolve 0.2 grams salicylic acid into 2.0 mL of methanol in a 5 mL round bottom flask.
- 2) Add 4-5 drops concentrated H_2SO_4
- 3) Add a few boiling chips to promote smooth boiling.
- 4) Reflux for 40 minutes.
- 5) After 40 minutes, remove and isolate product.

Discussion

In step 4, I used a hot water bath and not a sand bath, which is not the best method. The water vapours that come off from the water bath cause the condenser to heat up much too quickly, and some of the product vapour managed to escape the condenser. In addition, I didn't have a pipe cleaner handy (to wrap around the condenser to keep

Figure 1: Image of the initial setup (paper towel taken away for better image)



it cool) so I improvised and used some small strips of wet paper towel in an attempt to keep the condenser cool.

I also did not bother isolating the product (i.e. separating from the water, methanol that is left over, etc.) as I don't have the necessary reagents.

If you want to purify your product, you can do the following:

- 1) Pour the mixture into a test tube and add 4 mL diethyl ether.
- 2) Remove the aqueous layer with a (Pasteur) pipette.
- 3) Pour the organic layer into another (dry) test tube and remove any residual water.
- 4) Add 2 mL NaHCO_3 solution and mix. This will neutralize any remaining acid.
- 5) Transfer the ether layer to another (dry) test tube and add CaCl_2 pellets until the product is dry.

Figure 2: Image of the setup in the hot water bath (paper towel taken away for better image)



References

- [1] Williamson, Kenneth. Macroscale and Microscale: Organic Experiments. 4th Edition. Boston: Houghton Mifflin Company. 2003. pg. 486-497.