

IBX

50 g will yield ~40- 55 g of IBX

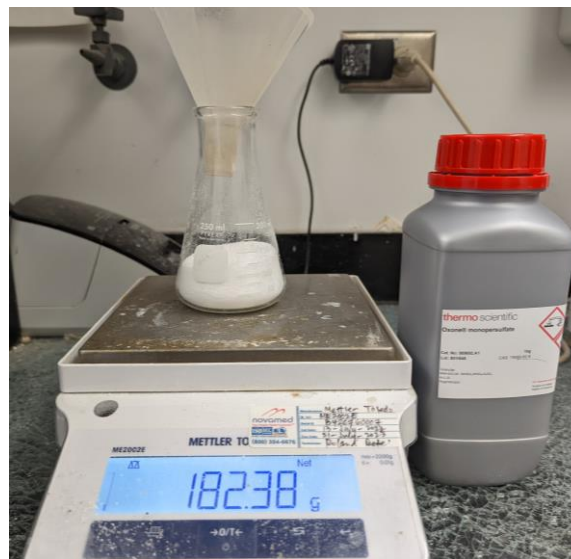
Caution! IBX is explosive at > 200 °C and is reported to be impact sensitive.

Waste is highly oxidizing and acidic. Treat with sodium thiosulfate and neutralize before disposal.

Santagostino, M. J. *Org. Chem.* **1999**, *64*, 4537-4538. <https://doi.org/10.1021/jo9824596>



Use a three neck RBF, equipped with a mechanical stirrer and condenser.



Weigh out 181 g of Oxone .



Dissolve Oxone in 650 mL of DI H₂O at r.t. will turn from cloudy to clear within 5 minutes



Weigh out 50 g of 2-iodobenzoic acid



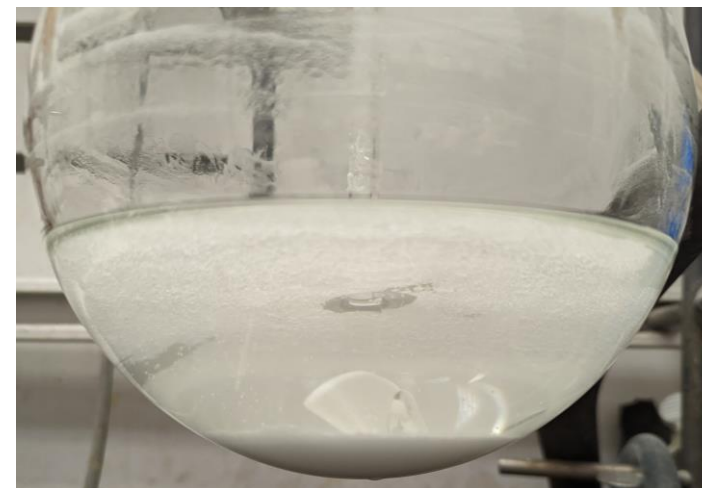
Add in a single portion, and raise temperature to 70- 73 °C



Starts off as a suspension with large particles. Increase the speed of the mechanical stirrer



The reaction mixture should become much finer in its consistency. The image shown is after ~2 hours of stirring and is a **fine solid with no solids stuck to the walls of the RBF**. The mechanical stirrer should be at or near the max setting, **without vigorous stirring this consistency is difficult to obtain**. After 3 hours at 70 °C the reaction is cooled. If stirring is stopped a fine white powder settles to the bottom.

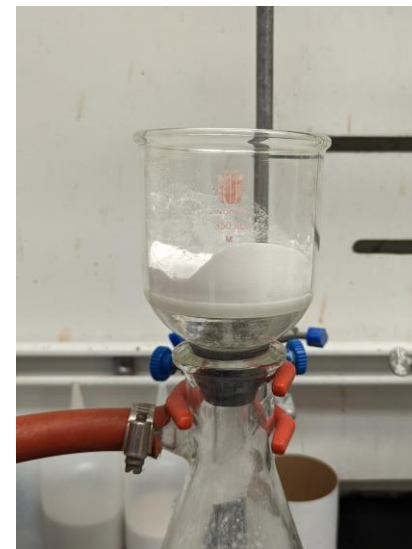




Cool the solution to 0 °C. Put the speed of the mechanical stirrer on the lowest speed, and stir for 90 minutes



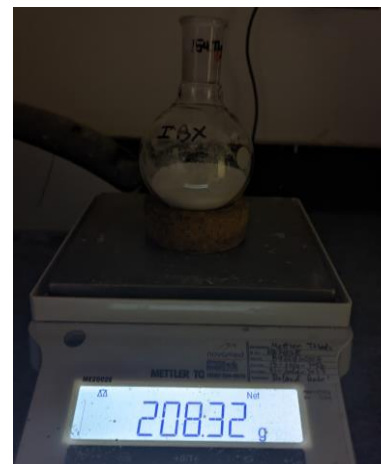
Filter to collect a fine white power
Wash with a total of 600 mL H₂O
and 200 mL of acetone
Washings are oxidizing and
acidic!



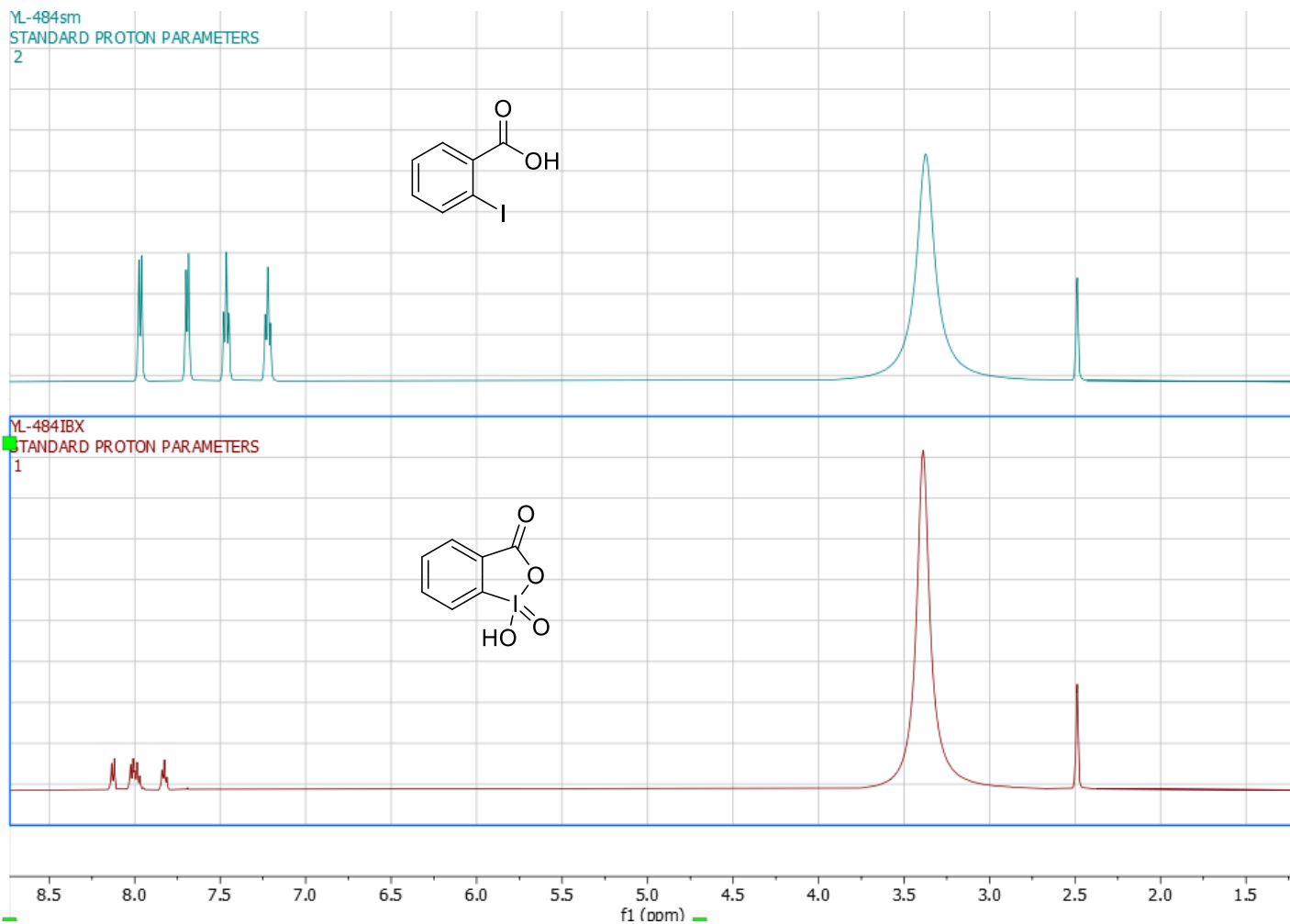
IBX!



Transfer to a RBF cover
in aluminum foil. Dry
under vacuum overnight.



56 g of IBX
95 % yield



IBX prepared matches literature,
 $^1\text{H-NMR}$ (DMSO- d_6):
8.15 (1H, d), 8.01 (1H, d),
7.98 (1H, t), 7.84 (1H, t).