

## DMP

**Caution!** IBX and DMP are heat and shock sensitive with exotherms as low as 130 °C reported. The preparation of DMP should be done behind a blast shield.

See the IBX preparation on our website

[https://www.sarlahgroup.com/files/ugd/f6a755\\_79985145b8f043f4a5cab233e68c8b7c.pdf](https://www.sarlahgroup.com/files/ugd/f6a755_79985145b8f043f4a5cab233e68c8b7c.pdf)

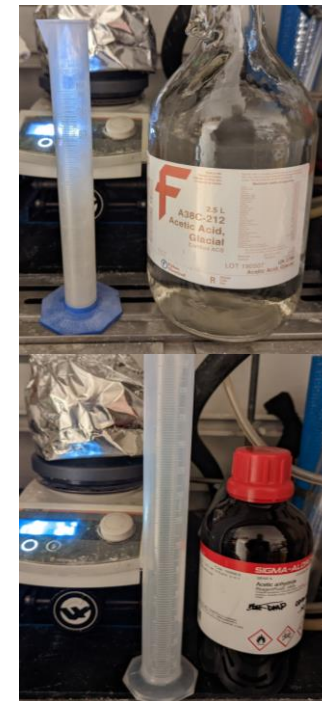
Mullins, J. *Org. Synth.* **2000**, 77, 141. DOI: 10.15227/orgsyn.077.0141



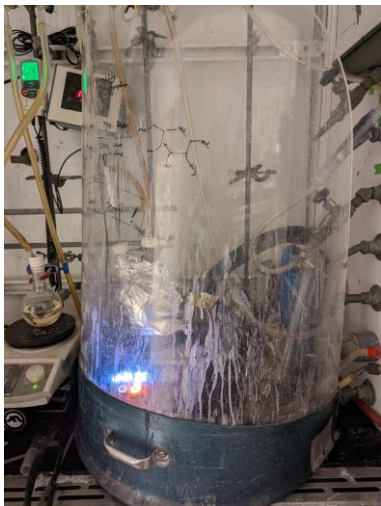
Use a 1 L two neck RBF, equipped with a temperature probe and a nitrogen line.



Add 56 g of IBX.



First add 95 mL AcOH then 190 mL of Ac<sub>2</sub>O.



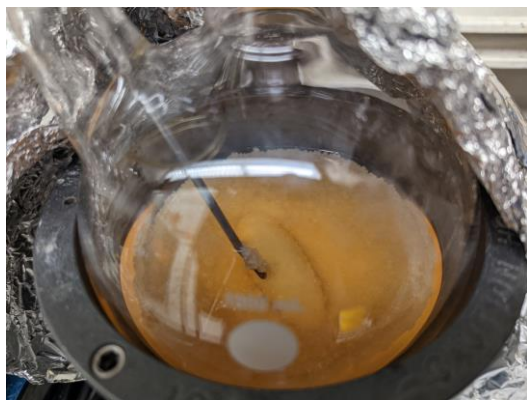
Place a blast shield in front of your reaction, then set the heating to 85 °C



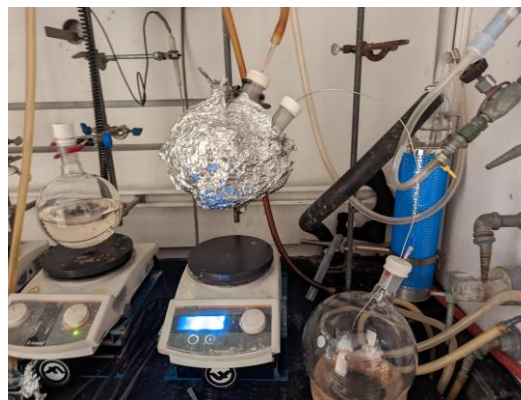
Initially the solution will be cloudy



After stirring for 20- 30 min (or until the solids dissolve) turn off the heating



Allow the solution to cool slowly over 24 hours. Large whit crystals will cover the bottom of the round bottom



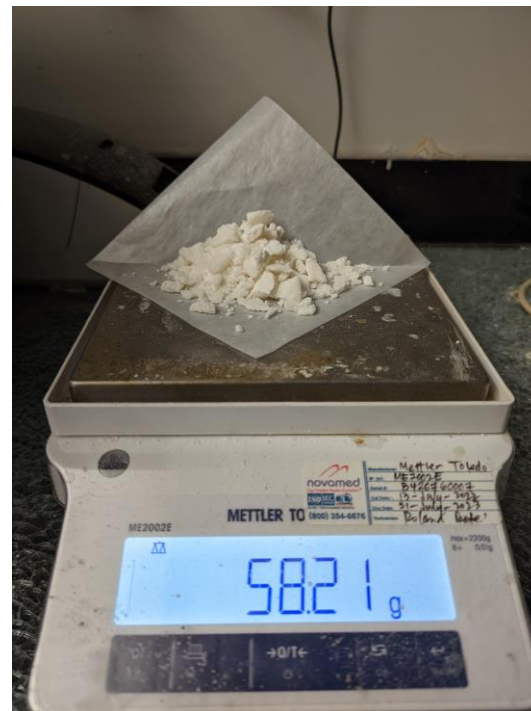
Use a canula to remove the mother liquor



Then add diethyl ether 40 mL, and canulate to remove. Repeat three times.

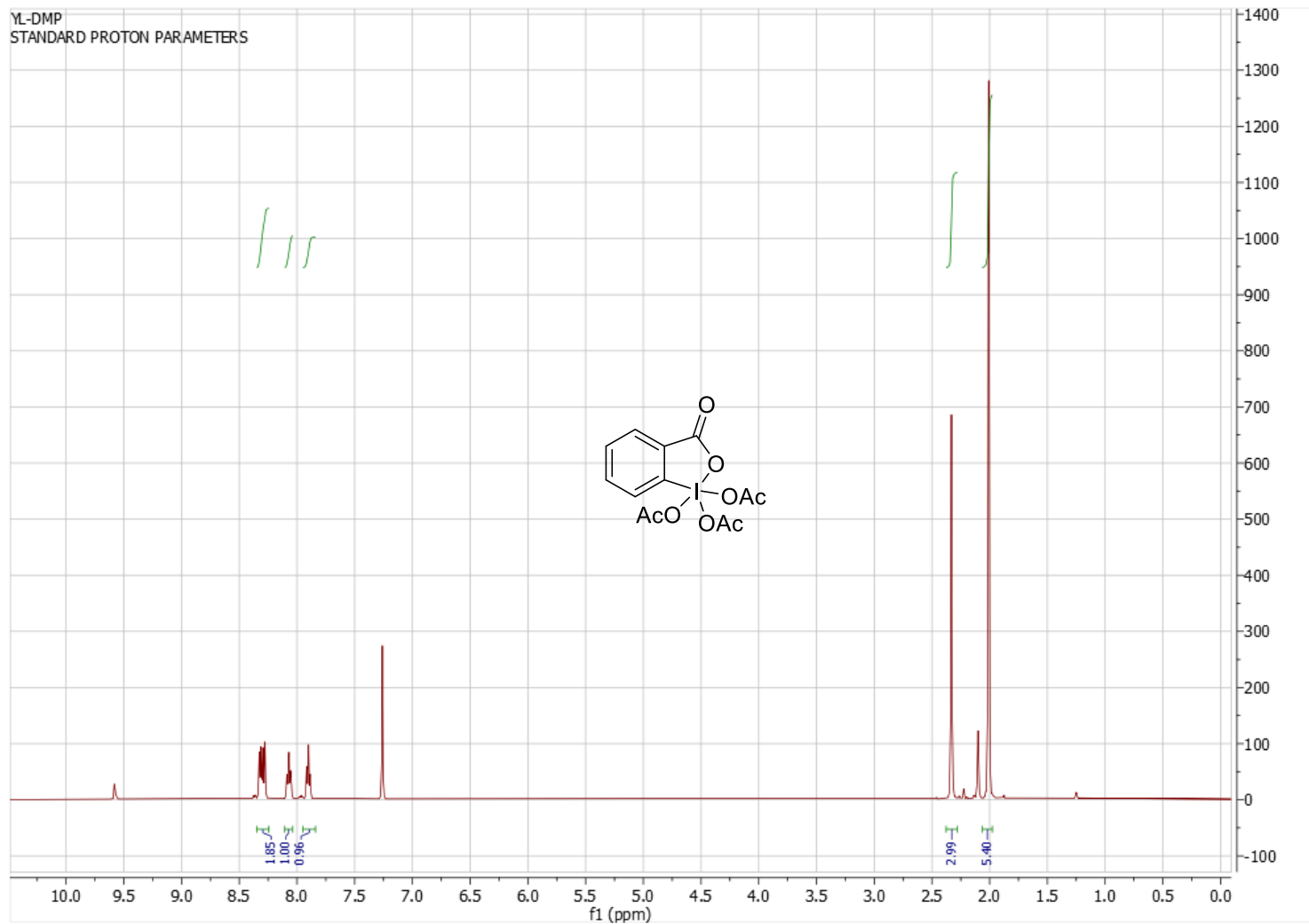


With all the solvent removed leave on the high vac over night.



This gave of DMP from 56 g IBX

YL-DMP  
STANDARD PROTON PARAMETERS



DMP prepared matches literature,  
 $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ): 1.99 (6H, s), 2.32 (3H, s), 7.91 (1H, t), 8.09 (1H, t), 8.29 (1H, d)