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METHYLAMINE FROM AMMONIUM CHLORIDE AND FORMALDEHYDE

A PHOTO-ESSAY

HTML by Rhodium

Many bees are making methylamine out of hexamine and HCl acid. I think it's very time consuming and HCl is pretty nasty. Thus I have found that one can easily make lots of good quality methylamine from ammonium chloride and formaldehyde (formalin). Only drawback with this method is that one must have good quality formaldehyde. It may have paraformaldehyde sediment in the bottom, it doesn't hurt, not even a bit, but Formaldehyde has to be strong and not some diluted shit, 35-40% is fine.

Now to the more-less foolproof method. If you do like said below, you get around 400-450 grams of pretty pure dimethylamine-free methylamine hydrochloride which is perfect for Al/Hg reductive aminations.

CHEMICALS NEEDED:

- 1100g of Ammonium Chloride
- 2 liters of 35-40% Formaldehyde
- about half a litre of chloroform

THE PROCESS



Pour your formaldehyde in a big round bottom flask (use at least one of 5 liter capacity) along with ammonium chloride and stir a bit. I used a 12 L RB flask. Start heating slowly, and stir it every once in a while.

[At 40°C] - [At 80°C]

At about 90°C, an exothermic reaction kicks in (it needs to be cooled in a water bath once in a while at the beginning in order to keep temperature below 104-106°C).



Soon reaction slows down and cooling isn't needed anymore, reaction flask must be heated carefully to keep temperature at 104-106°C.





Transfer it to a smaller flask and boil water away until you see the internal temperature rise over 160°C, then turn on the vacuum and continue, first temperature falls a few tens of degrees and when it rises back to about 160°C the methylamine is almost free of water. Turn off the vacuum and the heat.



Pour your crude methylamine hydrochloride in a beaker. At first it is still liquid (in this picture it has started to solidify, you can still see some orange liquid at the bottom of the beaker).



When it has cooled down, you'll have a big chunk of hard and dry methylamine. Wash it a few times with chloroform and you'll end up with a fully white mush.

Vacuum filter to get pure and dry Methylamine.HCl which reeks of chloroform. The smell can be removed by drying in a vacuum desiccator. Do not try to use the oven to get rid of these traces of chloroform, the Methylamine.HCl picks up water from air like nothing. You can only use vacuum for sucessful drying but these CHCl₃ traces don't hurt the methylamine at all and may be left in, but you might perhaps be a perfectionist...

