SYNTHESIS OF DIALKYLTRYPTAMINES (DMT & DET)

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DMT SYNTHESIS

STEP I

Using an area of good ventilation or a fume hood, place a 1000 ml two hole roundbottom flask in an ice bath using the setup in Figure II (you want a wobble stirrer in the top hole of the flask, and a separatory dropping funnel into the side entry). Add 400 ml cold anhydrous ether to the flask, in which 60 g indole is then dissolved, using the stirrer. To 100 ml anhydrous ether in a separatory funnel add 50 g oxalyl chloride. Slowly drip this solution into the vigorously stirred indole solution over a period of 10 to 15 minutes. Continue stirring 10 minutes longer. Allow the precipitate to settle a few minutes and decant the liquid. Add anhydrous ether and mix well. When satisfied as to the purity of the precipitate, leave the golden precipitate in the flask for the next step, which must follow immediately. Yield is approximately 100 g.

STEP II

Dimethylamine reacts readily with indole oxalyl chloride. Use about 400 ml ice cold anhydrous ether in the same 2 neck 1000 ml RB flask used in Step I, with the precipitate in it from Step I. Cool the ice bath further by using salt and ice. Estimate the weight of the precipitate and use 100 g indole oxalyl chloride. For this weight of IOC use two entire 50 g containers of diethylamine since it will not keep if the container seal is broken. Cool the amine in container much below 0°C and dissolve 1 part amine in 3 parts anhydrous cold ether. Amine may be stored in this solution. For use, warm stock solution to room temperature and use the appropriate aliquot. Set up the entire apparatus the same as when adding the oxalyl chloride. Add the amine solution slowly to the IOC with vigorous stirring. Stir for 1/2 hour after the addition is complete. Vacuum filter the precipitate, using ether as a wash. It is better to slurry the ether water with the precipitate before filtering [method used]. Recrystallise from hot ethanol or from a 50-50 methanol-benzene mixture.

STEP III

Prepare apparatus as in Figure II (1-hole 1000 ml RB flask set in heating mantle on magnetic stirrer with stir bar in flask, and condenser inserted into top of flask). Prepare the indole glyoxyl amide by melting and casting into sticks if ether is to be used as a solvent. Aluminium foil makes a good mould for casting pieces that will fit through the condenser. Also a Soxhlet extractor may be used to add the crystals by slow solution into the ether. Tetrahydrofluran, if available, dissolves IGA and the compound is added slowly in the solution form *[method used]*.

To a stirred mixture of 15 g $LiAlH_4$ in 100 ml anhydrous ether (or THF *[used]*) slowly add the sticks (or solution *[used]*) of IGA until 20 g have been added. Keep the rate of reaction at a reasonable rate or boil-over may occur *[do say!]*. Stir and reflux for 90 minutes after the addition is complete. Cool in an ice bath and begin to cautiously *[do say!]* hydrolyse with chips of ice or a cold solution of methanol, added through the condenser. When there is no further reaction, add a few ml extra water and allow to settle finally and decant the clear liquid into an evaporating vessel. Filter the residue and wash several times with ether- methanol or THF-methanol *[used]*. Evaporate the combined extracts and if necessary, seed the heavy syrup with crystals of DMT. With no seed crystals the product may take days or even weeks to crystallise *[weeks]*. This crude product is adequate for smoking *[do say!]*. In order to purify DMT, begin after the $LiAlH_4$ has been hydrolysed with methanol. Add 500 ml satd. Na_2SO_4 solution, mix and filter. Wash with ether or THF and neutralise the filtrate with 0.1 N HCl. Extract with ether in a separatory funnel

and neutralise the lower layer with 0.1 N NaOH, extracting this solution in turn with chloroform. The chloroform layer is dried over anhydrous Na_2SO_4, concentrated, and from it DMT crystallises on addition of petroleum ether. The mother liquor can be chromatographed on an alumina column using benzene-methanol in a 99.8 to 0.2 ratio. *[This last purification is quite difficult.]*

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DET Synthesis (Diethyltryptamine)

Step I

Same as for DMT.

Step II

Use 200g diethylamine per 100g IOC. Diethylamine is less volatile and poisonous than dimethylamine, so cooling is not necessary, but the fumes are poisonous. Use the same procedure otherwise. Diethyl derivative is easier to work with.

Step III

Use same procedure and equipment. Use 22g indoleglyoxyl diethylamide. The final product is also easier to purify.

Notes

Step I

Absolutely anhydrous ether is essential. A container that has been opened previously is no longer anhydrous. Where cooled reactions are necessary, remember that moisture is drawn to cold objects, and cold reagents, when left open or poured, become quite wet. This applies to the initial reaction on all three steps. A magnetic stirrer will not work for steps I and II. The vigorous wobble-stirrer has been found adequate do deliver the violent stirring needed, especially when several stirring balls are used in conjunction with the paddle-bar. Sparkless motors must be used around ether.

Oxalyl chloride is very toxic and ventilation or a fume hood must be used.

Water vapor hydrolyses product I, producing a gummy dark-red mass. Proceed to Step II as soon as possible.

Step II

Refer to notes on anhydrous ether, stirrer, sparkless equipment, and ventilation in the notes for Step I.

The color of the precipitate lightens somewhat as the amine is added to the compound I.

The water in the ether is used to dissolve all low-molecular-weight amines.

Step III

The crystalline amide is difficult to add to the $LiAlH_4$ mixture. A Soxhlet extractor may be used to add the amide by placing it in-between the flask and condenser. Casting it into rods or bars is one of the simplest methods. Tetrahydrofuran, if available, enables the indole glycoxal amide to be dissolved and added as a solution; a procedure which is best and fastest of all. $LiAlH_4$ is a very dangerous inflammable compound, especially so when in ether solution. The ether must be absolutely anhydrous or a violent effervescence occurs, destroying the $LiAlH_4$ and creating a fire hazard. Contact of $LiAlH_4$-ether solution with any water, damp materials, or even chemically bound water such as cellulose causes spontaneous combustion. A safety shield made from auto windshield material is a must when working with $LiAlH_4$ in any form. Handling $LiAlH_4$ is done wearing rubber gloves in a dry or inert

atmosphere with a minimum of friction involved. Hydrolysis of the complex is dangerous and should be done slowly and cautiously, using an ice-bath to cool the mixture.

Difficulty in producing crystals the first time should cause no concern since many organics need seed crystals to crystallize. The syrup may be used for some purposes but be sure to save some seed crystals if you should happen to get some.

Bibliography

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A Final Note (Apr 24 1995 -- Yogi S*/Lamont G)

The DMT recipe is misleading. Dimethylamine is a gas. DEA & DPA are liquids. DMA is typically sold as 40% solution dissolved in water. Since the indolyl reaction **must** be anhydrous, you will get absolutely *nothing* if you use the 40% solution. You'd either have to extract it into ether, and dry the ether. Or use the dry gas directly.

Beagle:

One small note regarding the use of anhydrous dimethylamine (or other amine) in the reaction with indole-3-acetyl chloride (or indole glyoxamide) to give N,N-dimethyl-indole-3-acetamide (or glyoxamide):

Surprisingly, the amine doesnt need to be anhydrous at all. Tests have shown that reaction of the acid chloride with 40% aqueous dimethylamine or dipropylamine gives equivalent yields of the amide as the reaction with anhydrous amine. There appears to be little to no indole-acetic acid produced in the reaction with aqueous amine.

Of course, the acid chloride is still extremely water sensitive, and scrupulous care keep conditions anhydrous during its formation and isolation are necessary to avoid unpleasant complex mixtures and low yields.