Purification methods for *d,l*-Amphetamine [salts]

This protocol describes two methods for the purification of d,l-Amphetamine salts (Better known as Speed): ***1.The acetone wash****; This will remove the caffeine from your sample and is also applicable on Cocaine, MDMA and Ketamine. The method can be repeated with Methanol to remove all of the sugars, Ketamine tends to have a lot.* ***2. Freebasing d,l-Amphetamine [salt] and recrystallization to d,l-Amphetamine Sulphate with sulfuric acid****. This will yield d,l-Amphetamine Sulphate with a maximum purity of 73%, removing all possible contaminants. The methods can be performed separately, but performing both in chronological order is preferred (The best would be to also perform the Methanol wash after the Acetone wash before continuing to step 2). In this protocol, d,l-amphetamine Sulphate is used for the calculations of the used volumes etc. This is because the Sulphate version is suspected to be the most abundant. However the groups can differ (e.g. d,l-Amphetamine Phosphate, d,l-amphetamine adipate, etc.), Therefore, Speed is described as d,l-Amphetamine [salt].*

Materials and methods:

Chemicals and solutions:

- *d,l*-amphetamine [salt] (s) (Speed) *(Method 1+2)*
- *d,l*-amphetamine [salt] solution (1g/10ml H_2O) *(Method 2)*
- 20% NaOH (w/v) (20 g/100 mL) *(Method 2)*
- Sulfuric acid solution in Acetone (30ml H_2SO_4 / 200 ml Acetone) (*Method 2)*
- Dehydrant ($MgSO_4$) *(method 1+2) (optional)*
- Acetone (anhydrous) *(method 1+2)*

Apparatus :

- 2x beaker (300ml) *(method 1+2) (clean glasses will suffice)*
- Coffee filters *(method 1+2)*
- pH meter (or paper) *(method 2)*
- Pasteur pipettes *(method 2)*
- separation funnel *(optional, method 2)*
- Funnel (to hold the coffee filter) *(method 1+2)*

Procedure

Preparation of chemicals:

1. [optional]: To obtain the dehydrant, spread Epsom salts ($MgSO_4 \cdot 7H_2O$) over aluminum foil and bake in the oven at 230°C for 1.5 hours.
2. Crush the product and heat again 230°C for 1 hour.
    - *Make sure it is stored airtight as this prevents hydration by H_2O from the environment*

3. Anhydrous acetone is prepared by adding an excess of dehydrant to the acetone.
    - *Do not pour anhydrous acetone when using it but pipette from the bottle. This prevents the dehydrant from ending up in your sample, as the excess of dehydrant settles at the bottom.*

4. Prepare the 30:200 (v/v) Sulfuric acid : Acetone solution right before using it as there appears to be color change from clear to dark red to black over time. If anyone knows why please let me know, I suspect it is either some non-influential decomposition or a reaction between the acetone and sulfuric acid which might affect the sample.

Purification method 1: Washing *d,l*-amphetamine [salt] with acetone

1. weigh the amount of your *d,l*-amphetamine [salt] and note it down. This is for yield determination.
2. Put 10ml anhydrous acetone/g *d,l*-amphetamine [salt] in a beaker and slowly add your *d,l*-amphetamine [salt]. Add small bits and stir very well after each bit is added.
3. Stir very well for several minutes. Make sure to break big clumps with a spatula or spoon.
4. Filter the solution through a coffee in a second beaker. The residue left on the coffee filter is your (still wet) purified *d,l*-amphetamine [salt]. Pour some more acetone over the obtained product in the filter for a higher purity.
5. Leave to dry and note weight difference. Determine % yield.
    - *Multiple washes are preferred to increase purity.*

Purification method 2: Freebasing d,l-Amphetamine [salt] and recrystallization to d,l-Amphetamine Sulphate with sulfuric acid:

1. Adjust the pH of the *d,l*-Amphetamine sulphate solution to ~12.5 using 20% NaOH solution (w/v).
    - *The d,l-amphetamine [salt] is now converted into amphetamine freebase and is now insoluble in water; amphetamine freebase is an oily substance, thus phase separation can be performed. However, the density of the freebase is almost equal to that of water, so it will take a while before two clear layers have formed. BE PATIENT!*
    - *Make sure to store airtight if you're going on a break since amphetamine freebase is volatile or continue to step 2 further purification.*

2. Extract the freebase (top layer) with a pipette or even better a separation funnel and dissolve in acetone with an amount of 20 moles solvent equivalent to 1 mole Freebase amphetamine (MW Amphetamine =135.21g/mol, MW Acetone =58,08g/mol).
3. [Optional] Remove remaining water by adding solid dehydrant and filtering the solution.

4. Dropwise add sulfuric acid in acetone solution to the freebase with a lot of stirring to convert the freebase into salt again until pH=5-6.
    - *Make sure the sulfuric acid in acetone solution is prepared just before using it. If the color of the solution has changed after a while, prepare a new one.*
    - *Be careful and patient! It is really easy to add too much and this will ruin your product.*

5. Filter solution. The residue is your purified product. Let it dry and determine yield.