Extract DXM From Cough Syrups and Gelcaps

I'm going to present this as "kitchen chemistry" as I feel most people with adequate chemistry knowledge (and equipment) will be able to do it correctly without my help.

There are three procedures for DXM extraction that are commonly used: precipitation and filtration, single-phase acid-base extraction, and dual-phase acid-bsae extraction (the "Agent Lemon" process). The first method is by far the least popular because the DXM precipitate is often so fine that it passes through the filter paper.

You can, of course, still use the precipitation procedure; I just don't recommend it. If you do choose to precipitate DXM, try to get actual filter paper rather than a coffee filter - it will help.

Theory of Acid-Base Extractions

The acid-base extraction process is a common method for isolating a desired chemical from undesirable "gunk". The theory is that certain chemicals (generally, alkaloids) occur in two forms: a water-soluble complex with an acid, and an oil-soluble free base form. For example, pseudoephedrine (Sudafed™), a decongestant, is usually supplied as the hydrochloride salt (pseudoephedrine HCl). It can also exist as a base, without an acid molecule (thus the term "free base"). You can convert an alkaloid from acid salt to free base (or vice versa) using a base (or acid).

The practical upshot is you take your chemical and "gunk", and raise the pH with a base (e.g., sodium hydroxide) until the chemical converts to free base form and precipitates out (since it's no longer soluble in water). Now you add a nonpolar solvent (an "oily" layer) for the chemical to dissolve in, shake for a long time, and all the chemical you want is in the nonpolar layer. Discard the polar (i.e., water) layer, and you're left with a nonpolar layer full of your chemical .....

Plus anything else that might be oil-soluble. So you reverse the process, by adding an acid until the free base turns into an acid salt, and precipitates out of the nonpolar layer. Add water, shake, and you can discard your nonpolar layer.

This is the acid-base extraction, and it's very frequently used to extract the active ingredients from plants (free clue: the THC in marijuana is *not* an alkaloid and thus won't extract this way).

Single-Phase Acid-Base Extraction of DXM

So how do we apply this to DXM? Well, it turns out that DXM is an alkaloid, and you can extract DXM from cough syrups using the same process. Furthermore, this procedure even works for DXM plus guaifenesin syrups, e.g., *Robitussin DM*, and generic equivalents (invariably called *Tussin DM*). The "DM" syrups usually only contain 10 mg/5ml of DXM, so you won't get as much yield, but they're usually cheaper (and more commonly available).

This is actually a single-phase acid-base extraction, because we only go from acid form (DXM HBr) to base form (DXM free base). The final product ends up dissolved in an organic solvent, which is then evaporated to leave DXM free base.

I have added a new set of steps to this process to help to remove some of the gunk that can end up in the final product. These steps are in italics and may be omitted if desired.

Do NOT try this extraction procedure with cough syrups or formulations containing acetaminophen/paracetamol, pseudoephedrine, other decongestants, or antihistamines. Decongestants and antihistamines are usually alkaloids and will end up in the final product; as for acetaminophen, I'm not convinced yet of the safety of the final product.

For this procedure you will need:

- Cough syrup (obviously) or some other DXM-containing preparation. The **only** active ingredients that should be listed are *dextromethorphan* and *guaifenesin*. Avoid alcohol

(check the inactive ingredients). If you can get DXM-only preparations, do so; the DXM+guaifenesin preparations tend to contain less DXM than the DXM-only ones.
- Two plastic two-liter bottles, washed and with the label removed. Of course, you can use flasks if you have them.
- A glass container to make your sodium hydroxide solution in (a mason jar works; you can also use a drinking glass).
- Two plastic bags with a slide-lock closure (e.g., Ziploc™), big enough to hold the cough syrup plus an additional amount of lighter fluid. The plastic bags should be non-pleated. They will be used as separatory funnels.
- A nonpolar solvent. The easiest to get is Zippo™ lighter fluid (or an equivalent) - note that this is *cigarette lighter* fluid, not charcoal lighter fluid. You want your solvent to evaporate quickly, leaving no residue. The easiest way to test it is by placing a drop or two onto a pocket mirror, and letting it evaporate; if it leaves no residue or smell, you can use it.
- Sodium hydroxide (NaOH). Photography supply stores carry this. In a pinch, some people have been known to use Red Devil™ Lye. **I do not advise this!** Lye is likely to be impure. If you must use lye, make sure you let your sodium hydroxide solution settle (see below). Note that sodium hydroxide is caustic and severely damaging to the eyes, so wear your safety goggles!
- A heat-resistant glass baking dish (smaller is better).
- Distilled water (tap water won't work as well due to the chlorine and dissolved ions).
- A pair of scissors
- Access to the outdoors.

To speed up the process (from overnight to about 30 minutes), you will have to evaporate the solvent with heating. For this you will require:

- An electric wok or skillet, or a hot plate with a pot of water on it. Basically, you want a flameless (electric) source of heat that will heat up a volume of water, which you can put your baking dish in (the hot water will heat the baking dish).
- A hair dryer
- An **OSHA-certified** organic vapor mask

Some warnings about organic vapors. The solvents you will in all likelihood be dealing with (hexane, heptane, petroleum ether, whatever) are bad for you. **Really** bad for you -- they can give you brain damage if you inhale too much of them. You do **NOT** want to breathe the fumes. Get it? So, if you want to speed up the process, pony up US$30.00 or so for an OSHA certified organic vapor gas mask (tell `em you'll be painting with oil-based paint). Sure, it's uncomfortable and looks dorky. But it sure beats brain damage! Additionally, you **absolutely must**do the evaporation outdoors (unless you happen to have a fume cabinet handy. And **NO**, the stove or bathroom fan does **NOT** count as a fume cabinet).

A brief word or two about sodium hydroxide: it's caustic, especially to the eyes, and when you add it to water it will heat up. Always add the sodium hydroxide to the water, and not vice versa. If you get it on your skin, wash it off with water (it won't eat through your hand unless you let it sit there).

If you can't find sodium hydroxide at your local photo store, go get a photography magazine and look in the back for mail order suppliers. Many of them carry sodium hydroxide. It is a very common chemical and ordering it isn't going to bring the DEA knocking down your door. Please try to avoid using lye. If you still can't get sodium hydroxide, use the Agent Lemon process.

Okay, here we go:

1. Form a solution of sodium hydroxide (NaOH) by placing one tablespoon (15ml) of solid sodium hydroxide in one cup (about 236ml) of distilled water in the sodium hydroxide solution container. Stir until dissolved. If you are using lye (I don't recommend it), wait awhile to let any impurities settle out to the bottom. Note that dissolving the NaOH will generate some heat.
2. Empty your cough syrup or formula into the two-liter, rinsing the last of the cough syrup out of the syrup bottles with distilled water. If using gelcaps, break them open and rinse out the inside of the capsules.
3. *The following steps in italics are suggested for removing some of the gunk that can make it through the extraction and leave you with a sticky residue instead of crystalline DXM.*
   Add in enough lighter fluid to the two-liter bottle to make a roughly 1/4 inch (or roughly 5mm) deep layer of lighter fluid per 4oz of syrup.
4. *Cap the two-liter bottle and shake the living hell out of it for at least five minutes. Let it sit undisturbed until the two layers separate again.*
5. *Pour the entire contents of the two-liter bottle into a sealable plastic baggie, and seal it shut. Hold it by one of the top corners so that a bottom corner is facing down. Let the layers separate again if necessary.*
6. *Holding the baggie's corner over a CLEAN two-liter bottle, snip off the very tip of the corner. Let the cough syrup layer drain into the clean two-liter bottle, but pinch it shut right before any of the lighter fluid drains out.*
7. *Discard the lighter fluid by placing it into an empty container and letting it evaporate outdoors. Do not put it down the drain or set it on fire.*
   That's it ... now you should have cough syrup that has had a great deal of the flavorings and other gunk removed from it. You can repeat the italicized steps if you want to remove even more.
8. Add one tablespoon (15ml) of sodium hydroxide solution to the two-liter bottle. You should see a rapid formation of a milky precipitate. Swirl the bottle gently to mix the syrup evenly, and the precipitate should redissolve (because there's not enough base yet).
9. Repeat the above step, until the precipitate *doesn't* redissolve with swirling. The entire solution should be cloudy (stir well to make sure the base is evenly distributed).
10. Add one more tablespoon (15ml) of sodium hydroxide solution to the bottle.
11. Add enough lighter fluid to make a 1/8" (0.3mm) deep layer per 4oz bottle of syrup.
12. Cap the bottle, shake the hell out of it for five minutes, and let it stand until the layers

separate again. If the layers don't want to separate, try adding table salt.

13. Carefully pour the contents of the bottle into the sealable plastic bag, and close it shut ("yellow and blue make green-it's sealed!"). Hold the bag by one of the top corners so that one of the bottom corners points down.
14. Let the two layers separate again (this should only take a few seconds).
15. Cut off the tip of the bottom corner and allow the water layer (the bottom layer) to drain out of the bag. When the water layer has drained out, pinch the bag shut.
16. Hold the bag over the baking dish, and allow the nonpolar solvent layer to drain out into the baking dish.
17. Take the baking dish outdoors. At this point, if you don't have a gas mask and a way to heat the baking dish, you'll have to let the solvent evaporate (which may take a day or so), so skip the next 4 steps.
18. Put on your gas mask and take the baking dish, hair dryer, and electric heat source outdoors.
19. Place the baking dish into the container of water (electric wok, electric skillet, hot plate with pan of water, whatever), and set it to simmer. If you can't set the temperature low enough, you'll have to turn the heater on and off manually to maintain a near-boiling temperature.
20. Plug in the hair dryer and *gently* blow hot air into the baking dish. Take care not to splash solvent over the sides of the dish. Incidentally, make sure you don't overload your circuit; it might be a good idea to alternate heating with the hot plate/wok/skillet and heating with the hair dryer.
21. Continue heating until all the solvent evaporates. At this point you may see a thin layer of crystalline material; you might see a shiny layer of goo that looks a lot like the glass itself (which can be confusing); or you might see a layer of brown gunk. Whatever. Anyway, make sure all the solvent has evaporated.
22. If your baking dish is covered with an oily substance (goo, gunk, whatever), you in all likelihood managed to extract some propylene glycol (or something else) along with the DXM. Blow hot air from the hair dryer onto the surface of the dish until the material dries completely (this may take 5 to 10 minutes). This should evaporate the propylene glycol, leaving behind only DXM.
23. Scrape the DXM off the baking dish with a razor blade or other convenient sharp edge. You now have DXM free base.

A few comments. First, guaifenesin seems to itself convert to an oily layer if you add too much sodium hydroxide, so don't overdo it. Second, if you happen to have lab equipment you can of course use a separatory funnel (which is what the plastic baggie is for). Third, if you don't think you got anything, make sure the baking dish is completely dry; sometimes the DXM free base plus propylene glycol can look a lot like the glass itself.

Agent Lemon: Dual-Phase Acid-Base Extraction of DXM

The Agent Lemon process is a newer and in all honesty a much better method for extracting DXM. It takes less time, doesn't involve playing with flammable and toxic fumes, and doesn't require sodium hydroxide. Here is the Agent Lemon method as posted on Usenet. I have inserted a few comments in italics, primarily with regards to separating the organic and water phases. They use a siphon tube, but I believe that the Zip-Lock™ bag separatory funnel is a better idea.

OPERATION AGENT LEMON
TOP SECRET

Reverend Jim Barris
Reverend Indole Ringh
Reverend Anastasia Albert

After establishing control over 80% of the world's Drixoral supply, our troop was in a position to change the face of DXMology forever, in the interest of accelerating human transcension.

The Mission: extract DXM from cough syrups with materials and equipment that one could buy without any trouble at your nearest Woolworth's.

Theory:

The DXM FAQ [1] describes an acid-base extraction method that requires materials (specifically Sodium Hydroxide) that are difficult to get in pure form for many people. Read it for the theory. Now, after you read it, we'll add on the following:

The "acid-base" extraction in the FAQ does not actually use an acid stage. We can add an acid stage,

to remove the DXM from the solvent. Therefore, we can avoid the need to evaporate a lot of solvent -- we can just throw it out the drain. Thus, you can do this without producing vapors that will lead your neighbors to think you are running a meth lab.

The Materials: (this will blow you away)

- DXM-containing cough syrup. This process will probably produce a dangerous product if you use a syrup that contains any active ingredients other than DXM or Guaifenesin. DON'T! We used Robo Max Cough because CVS was closed.
- Ordinary Household ammmonia (clear, not lemon or some other scent)
- Lighter Fluid (we used "Zippo"; check criteria in FAQ [1]). Make sure it evaporates with no residue.
- Citric Acid. Available as a canning supply at your neighborhood supermarket. We used lemon juice on the first attempt, but we switched to citric acid after consultations ([1], personal communication).

Equipment:

A brief interjection here. This paper suggests using a siphon to separate the layers. However, I think the plastic baggie separatory funnel is a superior method, for two reasons. First, rubber tubing (and many types of plastic) are attacked by organic solvents, and can degenerate, or (worse) dissolve into the solvent and possibly muck up the extraction. Second, you get much better control with a separatory funnel, even a kitchen chemistry version. I will follow up their method with my suggestion.

- Some containers and flexible rubber tubing to use as a siphon. We cut ours off our vaporizer because we don't know where we stashed the tubing.
- *Two large zipper-seal (e.g., Zip-Lock™) plastic freezer bags, unpleated (if you want to use the separatory funnel concept instead of the siphon).*

Concept:

Prepare ahead of time a solution of the citric acid in water. For two bottles of tussin (8 oz each) we used 3 tablespoons of citric acid in 8 fluid ounces of water.

Add ammonia to DXM. DXM converts from hydrobromide salt to freebase and precipitates out of water. Since it is now nonpolar it wants to go into a non polar solvent. Now you add a nonpolar solvent and shake hard. Free base goes into solution in solvent. Let solvent float to the top. (doesn't mix with water) Physically separate the layers. Now the DXM is in the nonpolar layer, mix that with the acid and shake well. The DXM converts back into the acid salt (since lemon juice has citric acid in it, we make DXM hydrocitrate). This is so beautiful because the DXM is practically pulled across the oil-water interface by the hydrogen ion gradient.

Now you throw out the oil layer, and the DXM is now acid salt in the lemon juice. Boil it for a few minutes in the microwave, stir it good, so any volatile solvent that remains will evaporate.

You are left with "Agent Lemon" or "DXemon juice", a highly concentrated product, which is superior to cough syrup -- if you really wanted to, you could probably boil away the water -- with no danger, since the amount of solvent is almost nothing, and get a crystalline product that might be cut with anhydrous citric acid. *I wouldn't suggest it, since it might irritate the stomach.*

Details:

1. Put cough syrup in 2 liter bottle.
2. Pour in a lot of ammonia. Excess is not a serious problem. The ammonia volume was about equal to the syrup volume.
3. Stir.
4. Pour into a tightly sealable vessel. A funnel is good to have.
   Incidentally, you can probably just use the same 2-liter bottle and pour the lighter fluid into that; the plastic won't dissolve.
5. Add about a 1/2 inch thick layer of lighter fluid.
6. Shake the hell out of the vessel. We did it for about five minutes.
7. Pour the liquid back into 2 liter.
8. Let the organic solvent layer separate, it wil float on top of the water.
9. We used a siphon to separate the layers. We filled the siphon (a flexible rubber tube) with water, and while covering one end, plunged the siphon deep into the water layer.

   Hold the free end of the siphon below the other end of the siphon, and let go of the end. Let the liquid drain into a jar. Throw out the watery layer.

   The other way to separate the layers is with a separatory funnel, or the approximate version (a plastic bag). Pour the entire contents of the bottle into the sealable plastic bag, seal it, let the layers separate, clip off the bottom corner, and let the watery layer (on the bottom) drain out into the drain. Then let the organic layer drain into the jar.
10. If you want to minimize the amount of water-ammonia-cough syrup inactive ingredients, add more water, let separate and siphon *(or separate)* again. We were paranoid and did this four times. The product we got towards the end didn't even taste like ammonia.
11. Now mix the solvent layer with the citric acid solution.
12. Transfer to snapple jar.
13. Really shake the hell out of it. We shook it for 5 minutes, splitting the work between the three of us.
14. It takes a few minutes to separate. Wait.

    Some have reported a soapy layer forms in between the two layers; if so, just let it sit until the soapy layer is completely gone (it may take a few hours).
15. Next stage. Use the siphon again, we recommend being conservative and not letting any

solvent get into the siphon at all, so we left a little water layer.

Or use a new plastic storage bag, and this time keep the water layer and discard the organic layer. Be friendly to the environment and let it evaporate outside; don't just pour it down the drain.

16. Boil the lemon juice for a few minutes (we did for seven), the theory is that if a little bit of the organic solvent is there, it will boil away.
17. Drink the Agent Lemon. We have not deterimined the best method, we think you could mix it with something, or maybe drink it straight. It tastes really bitter (that's the DXM).

Results:

(Note: This was a previous trial with 4 oz. of syrup and with lemon juice instead of citric acid. This may give an inferior product.) One of us (I.R.) volunteered to assay the material extracted. Since I am a 105 kg. male in good health (except for the tail end of a nagging cold) I was considered to be the best subject. I thinned the material from about 1 fl. oz. to about 8 fl. oz. and added six tablespoons of sugar. I consumed the material at approximately 4:20 in the afternoon. The extracted product seemed to have a washed-out lemon taste (some flavor oil probably went into the organic phase). There was a bitter taste which I believe is DXM. I tasted (very carefully!) a few microliters of the lighter fluid, and that didn't seem to match the weird taste.

After about 15 minutes (while the other investigators smoked a bong) I noticed significant pharmacological effects. At about 5:30 I was experiencing effects consistent with a dose of about 3.0 mg/kg. This suggests a yield in the 90% range. (about 315 mg of 350 recovered). At about this time, I took a couple of small bong hits myself, which produced instant second plateau effects. This backs up my assay of the dosage. At this time (9:30) I am still experiencing light effects.