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Medical Marijuana Solvent Extraction Efficiency – Potency Determinations with GC-FID

August 3rd, 2011 by Jack Cochran

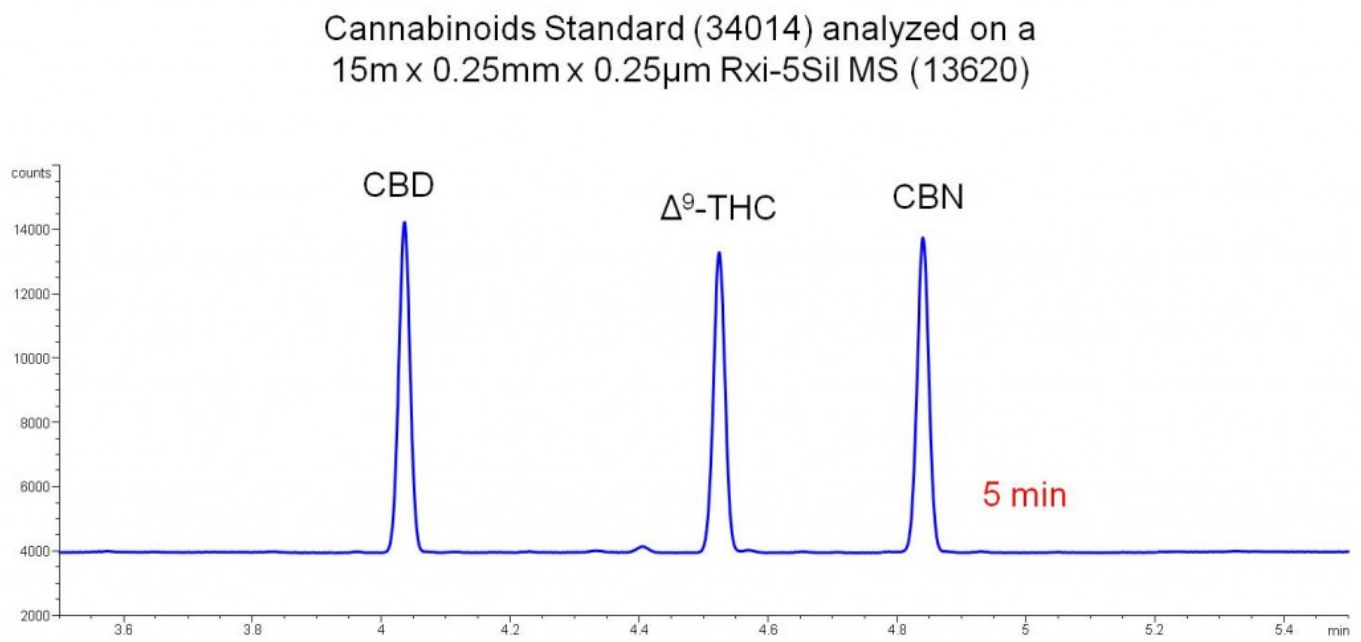
Amanda Rigdon and I recently investigated the extraction efficiency of various solvents for medical marijuana potency determinations (although technically we're not working with medical marijuana; instead we use seized illicit marijuana and did the work under the auspices of the Penn State University Police Department with Randy Hoffman, an Evidence Technician there).

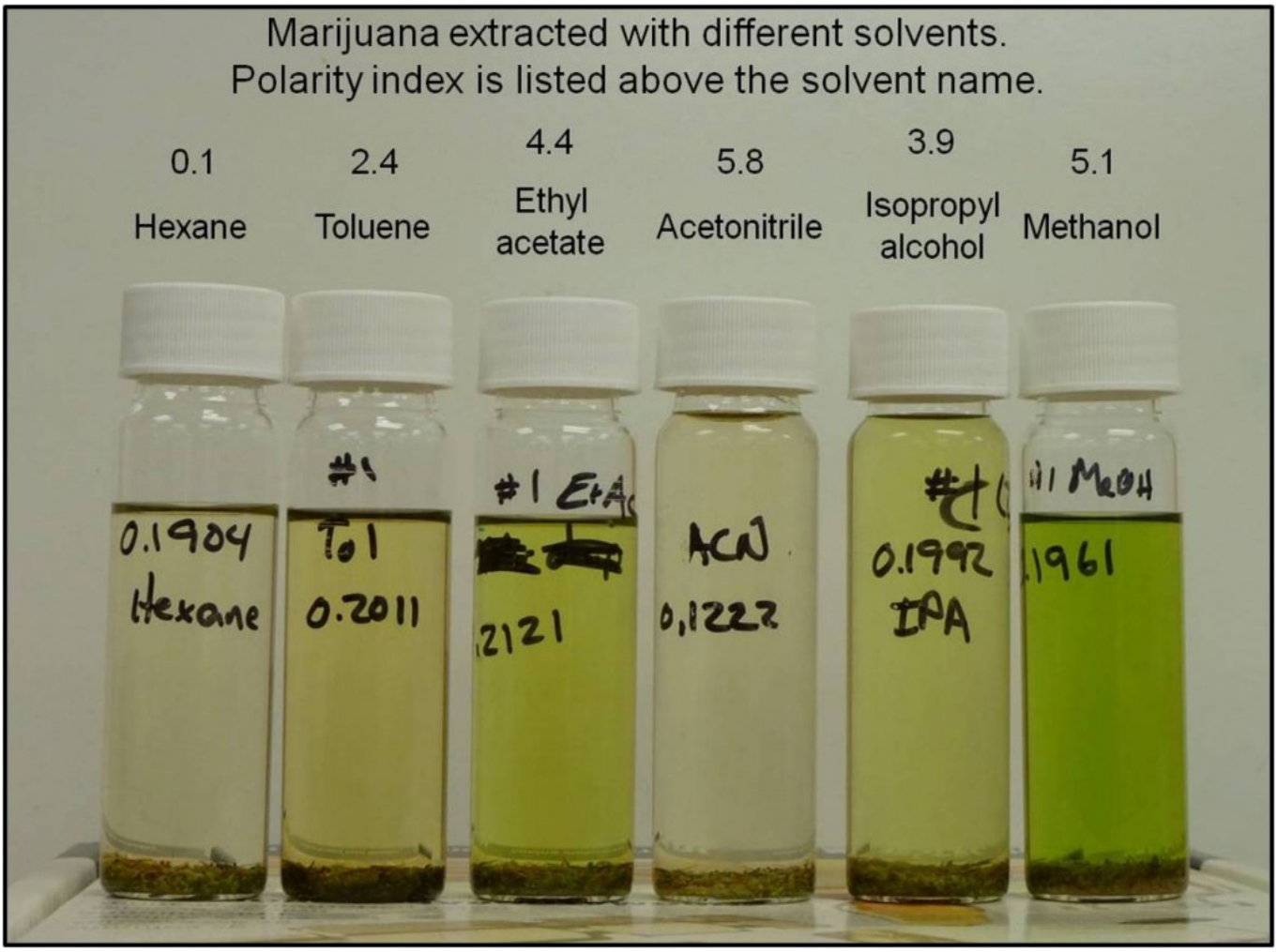
We used an Agilent GC-FID with split injection on a Restek Premium 4mm Precision Liner with Wool and a 15m x 0.25mm x 0.25µm Rxi-5Sil MS. A cannabinoids standard containing cannabidiol, delta-9-THC, and cannabinol was used for calibration and quantification. The last eluting compound of interest, cannabinol, had a retention time of about 4.85 min.

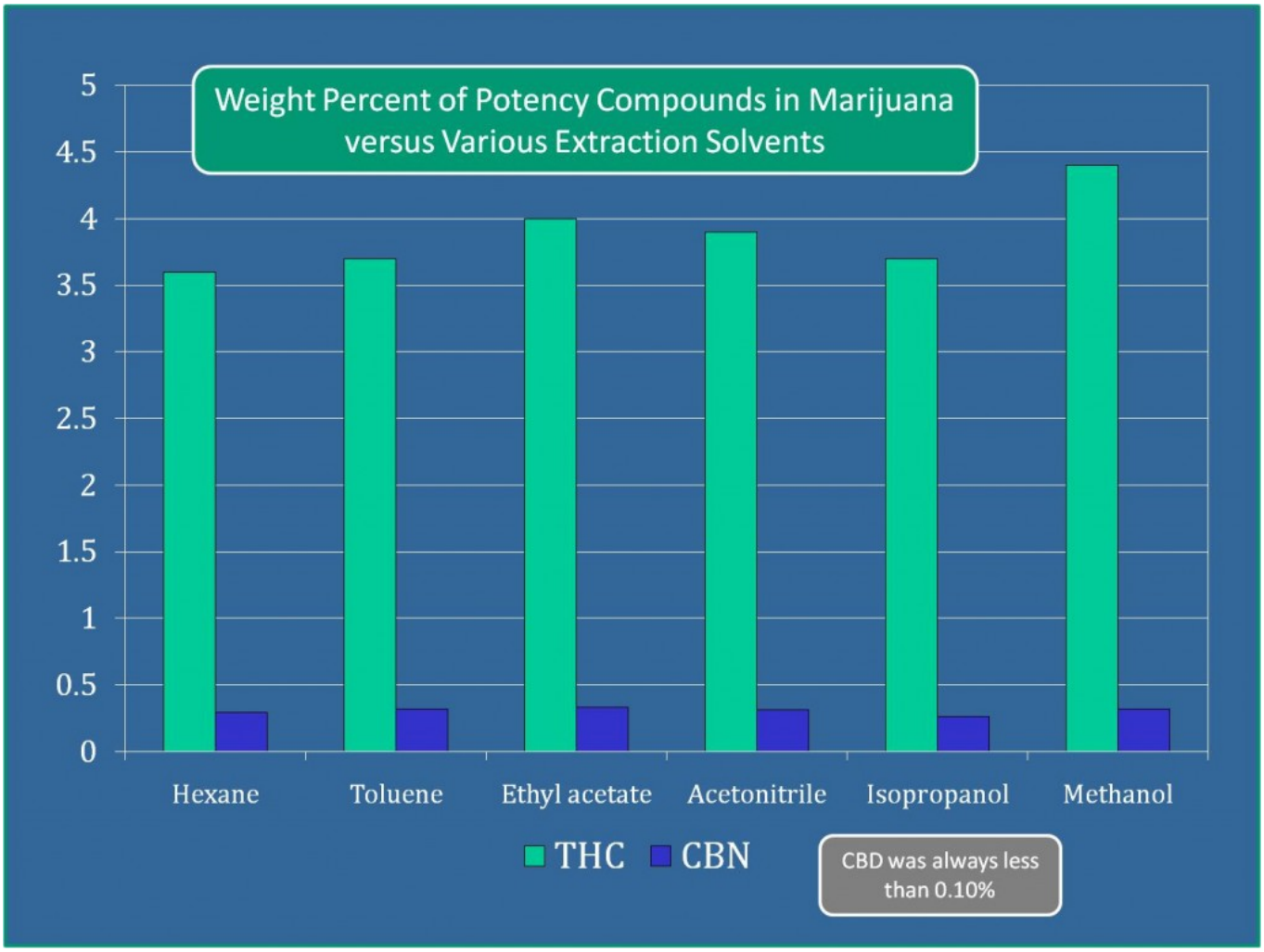
One consideration for extraction efficiency in marijuana potency determination is that THCA is the main source for THC determined with GC. (THCA, or delta-9- tetrahydrocannabinolcarboxylic acid, decarboxylates during smoking, or other heating, including in a hot GC inlet, to delta-9-THC.) Solvent polarity index (higher value means more polar solvent) was used to assure a range of tested solvents from non-polar (hexane) to highly polar (acetonitrile and methanol) as we tried to achieve maximum extraction efficiency of THCA from marijuana. Approximately 0.2g ground samples (except in the case of acetonitrile where we ran out of marijuana and only used just over 0.1g) were extracted with 40mL solvent in a precleaned VOA vial.

As seen in the bar graph, methanol was the most efficient extraction solvent for delta-9-THCA (analyzed as THC). Generally, the delta-9-THCA extraction efficiency correlated with solvent polarity, although not dramatically so. We had hoped that the non-polar solvent, hexane, would be equally efficient as the polar solvents for THCA extraction for one very important reason that can be seen in the photograph of the extracted samples, and that is, reduced extraction of chlorophyll by the non-polar solvent. Chlorophyll, a non-volatile substance providing the green color to the extracts quickly degrades GC inlet and column performance and could lead to erroneous potency determinations over time without timely GC inlet maintenance.

More later on the LC analysis of THCA from my colleague Amanda…


Cannabinoids Standard (34014) analyzed on a
15m x 0.25mm x 0.25µm Rxi-5Sil MS (13620)
counts
14000
12000
10000
8000
6000
4000
2000
CBD
Δ9-THC
CBN
5 min
3.6
3.8
4
4.2
4.4
4.6
4.8
5
5.2
5.4
min


Marijuana extracted with different solvents.
Polarity index is listed above the solvent name.
0.1
Hexane
2.4
Toluene
4.4
Ethyl acetate
5.8
Acetonitrile
3.9
Isopropyl alcohol
5.1
Methanol
0.1904
Hexane
0.2011
ACN
0.1222
0.1992
IPA
.1961



This entry was posted on Wednesday, August 3rd, 2011 at 4:10 am and is filed under Cannabis, GC Injection Techniques, Optimizing Applications. You can follow any responses to this entry through the RSS 2.0 feed. You can leave a response, or trackback from your own site.

34 Responses to "Medical Marijuana Solvent Extraction Efficiency – Potency Determinations with GC-FID"

1.  *Jamie Hart* says:
August 11, 2011 at 11:21 pm

   Hi Jack,

   You did not mention what you did for the extraction. Did you sonicate, heat, or simply shake?

   Jamie Hart

2.  *Jack Cochran* says:
September 11, 2011 at 12:01 am

   Hi Jamie:

   The extraction procedure was simple shaking for 5 min and then the sample was allowed to settle so that an aliquot of extract could be withdrawn for GC and LC potency testing. We have an article coming out in the

next Advantage with additional details. Be sure and watch for it!

Jack

3. *Tim Osborn-Jones* says:
January 9, 2012 at 10:27 pm

Hi Jack,

What type of grinding did you use to grind the samples to the fine powder prior to further extraction?

Tim

4.  *Jack Cochran* says:
April 20, 2012 at 10:24 pm

Hi Tim:

We used a simple, small Bamix food processing wand (with the dry ingredients attachment) to do grinding of the medical marijuana, but this is not something for larger scale work! Too time consuming, but we typically don't have large numbers of samples to grind and the Bamix does a good job.

Regards,

Jack

5. *Kurt Kramar* says:
June 24, 2012 at 8:15 pm

Hi Jack,

what percentage of isopropyl alcohol, were you using for the extraction process? reading all of the footnotes I didn't find the exact percentages. Was it 100%, 70, 50 or does it matter?

6.  *Jack Cochran* says:
June 26, 2012 at 11:30 am

Hi Kurt:

Great question, and I should have been more explicit. The isopropyl alcohol used for the medical marijuana extraction efficiency work, as well as all the other solvents, was indeed "100%", or a higher purity analytical grade of solvent. "Rubbing alcohol", aka 70% isopropyl alcohol that you can buy from the local drug store (the "chemist" in some parts of the world), is 30% water, and we typically avoid water when doing GC work. It brings up an interesting point though in that we often increase extraction efficiency for pesticide residue testing by pre-wetting our dry samples (even marijuana) with water prior to doing extraction. That doesn't seem to be necessary for cannabinoid testing however and avoids the problem of having to "get rid" of the water prior to GC analysis.

I'm wondering now after your question though if we shouldn't consider evaluating solvent:water as an extraction mechanism for cannabinoids, considering that we use a split injection for GC analysis of cannabinoids, which reduces the impact of water on the analysis. Thanks for the question!

Jack

7. *Chris* says:
July 9, 2012 at 5:25 pm

Medical Marijuan and MMJ products can be extracted with 91% IPA as commonly found in drugstores, or denatured alcohol found in a hardware store. Running comparisons of ETOH. MEOH, and IPA showed little difference in extraction. The issues with 912% IPA are mostly viscosity, and the ability to have good precision injection to injection because of the miniscus in a 10ul syringe is more difficult to read. Water is not an issue when using a .53 um collume for simulated distillation in the above extraction solvent cases listed.

Using ETOH is best case however expensive by comparison. Given the precesion of the test given non-standardized protocols across the industry and being subject to non schedule 1 cannabinoid standards for calibration which have run to run variations on product I have received from one manufacturer which can be as high as 30%, the solvent extraction efficiency of the three listed become less of an issue towards both accuracy and precision, than the lot to lot variations in calibration standards available.

CAT LABS

8. *Chris* says:
July 9, 2012 at 5:42 pm

Sample prep for flowers is done with good results by hand selecting pieces working from the apical meristems down pulling off three to 6 bractiols at a time and droping them into a pre tared 40 ml VOA vile. Weigh out in the vial 100-200 mg of flowers in this manner,. add your extracting solven and shake. Gentle heating for 5-20 minutes at < 50 degrees C results in .2 to .5 % by weight increase in value of d 9 THC, by increasing solvating efficiency.

Sample prep for hashis, ground to small particles, followed by extraction and heating. Use a clean wooden chopstick, and disturb, crush and remain large particulates, return VOA vial cap shake and then let settle befor injection.

Edibles start at 200 mg, 300 500 1G an up, extract with solvent disturb with a chopstick add MFSTA and pyridine 100 ul each to a mixing vial and inject into extract to derrivatize triglycerides that are found in most edibles. These triglycerides can bind to the sim dist collumn destroying its seperation efficiency

9.  *Jack Cochran* says:
July 25, 2012 at 12:47 am

Thanks for your comments, Chris! Much appreciated. Good point about minimal (to no) impact of water on your 0.53mm column; it has the capacity to handle some water (and other polar solvents). Are you using split injection?

Your comments on solvent expense make me wonder if we should be “micro-ing” the whole procedure, i.e. reduce the sample size and the extraction solvent volume. We do that in other fields quite successfully, with the caveat that you have to respect “sample representativeness”. Does the analyzed result from that smaller sample size represent the “true” value for the “complete” sample?

Jack

10.  *Jack Cochran* says:
July 25, 2012 at 12:50 am

    Many thanks for your sample prep education, Chris! Much appreciated. Interesting approach to preserving column efficiency (and lifetime) by derivatization of triglycerides! But why isn't the THCA (the acid that decarboxylates to THC in both heating/smoking and a hot GC inlet) converted to the TMS derivative by MSTFA?

    Jack

11.  *Aaron Letailleur* says:
January 10, 2013 at 5:17 pm

    Hi jack,
    I'm sorry for commenting on such an old post but I was just wondering where you think pentane would fit in on the graph. As far as I'm aware the polarity index of pentane is 0.0 so I was thinking would that make is more efficient? Or would it make a negligible difference?

12.  *Jack Cochran* says:
January 11, 2013 at 2:41 pm

    Hi Aaron:

    I'm glad for any and all comments, even on old posts. And it's not that "old", as regards science, because I'm getting ready to post some interesting stuff on GC column selectivity for marijuana potency analysis. But back to the subject… Pentane is extremely non-polar, as you note in your comment, so I expect its performance would be similar to hexane, which gave the lowest recovery for the THC acid (converted to THC upon hot split GC injection). One knock against pentane in this case, independent of extraction efficiency, is its volatility. Similarly to methylene chloride, it has such a high vapor pressure that it tends to evaporate while sitting in the vial on the autosampler tray and that could change the determined result. In this situation, an internal standard of some sort should be added to improve quantitative results.

    Thanks for the comment, Aaron.

    JC

13.  *WeedScientist* says:
May 9, 2013 at 5:16 pm

    I'm surprised to see so much CBN in your comparative analyses. Have you tried splitting your sample and running on HPLC? I have a feeling your CBN number is actually just representative of thermal degradation of the THCA/THC.

14.  *Jack Cochran* says:
May 10, 2013 at 2:17 am

    Great observation, Rose. You have to remember that because PA is not a medical cannabis State that any work we do is with marijuana that's been confiscated by the police, which means it's been stored in (ambient temperature) evidence lockers for extended periods of time, which leads to degradation of THC to CBN (or the acid to the acid).

Thanks for posting.

JC

15. *ed van der wal* says:
June 14, 2013 at 9:19 am

Hi Jack,

I was very pleased to see your blogs on cannabis testing. We do the same and have of course the same problem of the very difficult matrix. So the solvent experiments looks nice, at least the colour of the extract is different, with acetonitril being a possible good choice. However, the colour does not always is a good indicator for the problems during analysis (dirt on the GC -liner and peaks that can potentially mask the components you are looking for). It would be nice that life is so simple!
Can you say something about the effect of the different solvents on the troubles in the GC linear and do you have examples of the GC TIC chromatograms from the different solvents? That would be very much appreciated.

Thanks or all the good and interesting work

Ed

16.  *Jack Cochran* says:
June 14, 2013 at 5:37 pm

Hi Ed:

Thank you so much for your kind comments on our work. I completely agree with your assessment that color is not always a complete indicator of the problems that can arise during GC analysis, including “dirt” and matrix. I define “dirt” as something nonvolatile that sticks to the liner, the liner packing (if any), and the head of the GC column, while “matrix” I define as something that chromatographs and can interfere with peaks of interest. I guess technically “dirt” is “matrix”, but it doesn’t interfere as a peak that directly interferes with the peak of interest, in my definition. Dirt, however, is deadly on gas chromatography, as it can quickly foul the liner and lead to suppression of analyte response, or in some cases, degradation of analyte (e.g. DDT is a good example).

In the specific case here, extraction of cannabis, chlorophyll is providing the color and is a notorious GC inlet liner “killer”, so less chlorophyll is always better, if it doesn’t come at the cost of poor analyte-of-interest extraction efficiency. So in this case, while I don’t have the experimental data in hand, I think I can safely say that the solvent effects on the liner and column will be negligible versus chlorophyll buildup in the liner. One of the ways we can mitigate this “dirt” effect is to use high split ratio injections while meeting detectability requirements. The high flow through the inlet will help suppress many inlet issues that can arise for this type of work.

I hope this was helpful, and again, thank you for your comments. If you have any suggestions for other cannabis testing experiments, please let me know.

JC

17. *WS* says:
October 8, 2013 at 9:07 pm

Your excellent research has inspired me to ask a few questions.
Have you considered running an HPLC test (if you have access) to eliminate the thermal degradation that occurs due to the heating process involved in GC? That way you can run an analysis using the different solvents and compare the ratio of Cannabinoid acid levels to the Cannabinoids present after each solvent extraction.
Did you ever get results for using water as the solvent (hot (160 degrees C) or cold (-50 degrees C)?
Have you tried employing acidic water (pH 2.5) or alkaline water (pH 11.5)?
Also, have you considered running a double solvent extraction to minimize the levels of chlorophyl present in the extract? For example, extracting with methanol followed by hexane (to remove the chlorophyl).
Last question, have you considered employing different thermodynamic properties to the experiment. For example, utilizing very cold or hot temperatures for the solvent or biomass (below the degradation temperature for the cannabinoid acids/cannabinoids).
Thanks for your outstanding research, it is greatly appreciated!

WS

18.  *Jack Cochran* says:
October 8, 2013 at 10:02 pm

Great questions, and thank you for reading.

LC is certainly the way to go for analyzing the cannabinoid acids (e.g. THCA, CBDA, etc.) directly without trying to decarboxylate them via a hot GC inlet, although historically cannabis potency is determined using GC-FID. We have an article on looking at THCA via LC here in our Advantage newsletter:

http://www.restek.com/pdfs/GNAD1232-UNV.pdf

I think your suggestion for investigating different solvent extraction efficiencies for acids with LC is good, but ultimately if we're going to determine the acids via LC, we need to be in an LC-friendly solvent, for reverse phase, and that means something water miscible (e.g. methanol, acetonitrile).

I have not done any water extractions, but I'm definitely intrigued by your comments, especially for pH adjusted extractions. What would you expect?

The suggestion for doing back extraction with hexane to remove chlorophyll is also very interesting to me, and I will try that next time to determine if that can provide some basic sample cleanup that will result in longer uptime for the GC system. I've seen cases where later eluting cannabinoids (e.g. CBN) start falling off in response after sample analyses and those responses almost always come back after changing the GC inlet liner and bottom seal, and trimming a loop (about 0.5m) off the front of the GC column. I'm relatively sure that the chlorophyll is being "taken out as trash", so to speak, in that scenario.

I think hot solvent extraction, or any other vigorous means of extraction (e.g. extended shaking), has the potential to yield higher numbers (i.e. higher recoveries) for the analytes of interest, so that's another good observation you've made. When we revisit this work on extraction, I'm definitely going to refer to your comments again. Many thanks for them.

JC

19.  *T* says:
February 10, 2014 at 9:53 pm

Is there a non alcoholic solvent which can be used to get an efficient extraction? If so please name it

Searching for a yr now please help

TS

20.  *Jack Cochran* says:
February 11, 2014 at 3:13 am

Hello TS:

I'm not sure I understand your question, but hexane, toluene, ethyl acetate, and acetonitrile are all "non alcoholic" solvents that potentially could be used for efficient extraction. Methanol and isopropanol were the only alcohols used in the study posted. In general, all of these solvents gave relatively good extraction efficiency based on cannabinoid determinations via GC.

I hope this helps.

Regards,

Jack

21.  *VA* says:
April 6, 2014 at 6:28 am

Hi,
Based on the bar graph showing all of the weight percentages between 3.5 and 4.5, I have to ask, did you have controls to prove that the assay worked for solvents with efficiencies below or above that range?
Thank you

22.  *Jack Cochran* says:
April 8, 2014 at 2:28 pm

Great question! The quick answer is no, and is highly driven by the fact that Pennsylvania, where Restek resides, is not a medical cannabis State. We worked under the supervision of the police and Penn State University to do our pesticides and cannabinoids work on confiscated illicit marijuana, but by necessity (and law!), that work is limited in scope.

Caveat above aside, I expect similar extraction performance for higher (and lower) cannabinoid content, at least on plant material, especially flowers. However, I do think it would be of great value to the industry eventually to do an exhaustive-extraction study with various solvents across a range of materials.

The other thing we've ignored, and maybe it's even more important, is "sample representativeness". For example, when determining pesticides in food, some labs use kg amounts of strawberries, homogenized, to produce a representative smaller sample for extraction, i.e. avoid reporting very high or very low numbers by using only a few strawberries, which may contain more or less pesticides based on the spraying. My question for the medical cannabis community, at least for plant material, is how representative is the potency value obtained by using a small amount for extraction? That may be a moot question given that it's not practical to sacrifice large amounts of medicine for testing. And, I assume with edibles production the dose is more accurately controlled anyway.

Thanks for your question.

JC

23. *Josh Alper* says:
May 28, 2014 at 5:44 am

Do you have any numbers for ethanol as a solvent. 190 proof.

Looking for data on quick wash with ethanol, efficiency of quick wash of decarboxylated material to extract active ingredients without water soluble components.

Thanks for posting the things you have.

Josh

24. *Ahmad* says:
June 27, 2014 at 7:58 pm

Hello,

Quick question for you, don't know if I missed it but wondering what column and material of the column that you are using in the GC? Another question, do you know the amount of or concentration odd chlorophyll present in these samples? Lastly, if you were going to continue to remove the chlorophyll, have you thought of an additional filtering setup or quecher.

25.  *Jack Cochran* says:
June 27, 2014 at 11:48 pm

We're using the Rxi-35Sil MS or the MXT-35 for potency determinations via GC-FID these days (we used something different in this post, but it was a much earlier post). If you have an SRI GC, the metal MXT-35 column will fit in the smaller oven. If you have an Agilent GC, or other GC, consider the fused silica Rxi-35Sil MS. If you search on the blog you'll find helpful posts on the use of these columns for cannabis potency determinations via GC-FID.

Great question on how much chlorophyll is present in these extracts and the quick answer is, "I don't know!" But they can be very green depending on the amount of sample and solvent used for extraction. Generally, as long as your sample representativeness requirements are met and your limits of detection for components of interest can be met, you can minimize the sample extracted to keep the GC system up longer. Also, using split injection with the proper inlet liner is a great way to extend column lifetime and system uptime, too. There are some posts on that on ChromaBLOGraphy, also.

Removing chlorophyll can be done with graphitized carbon black via a dSPE technique, but I'm worried that you might lose some of the cannabinoids during that cleanup. You've got me motivated to do a project on it, though. I'll see if I can squeeze in some experiments in the next month or so.

Thanks for reading!

JC

26.  *Jack Cochran* says:
June 27, 2014 at 11:51 pm

Hi Josh:

I do not have any data for 190 proof ethanol as an extraction solvent for cannabis for potency determinations. Not sure what you mean about water-soluble components, but would expect ethanol to extract polar compounds (which generally have higher water solubilities) relatively efficiently.

JC

27. *Eric Winterstein* says:
December 15, 2014 at 6:45 pm

Hello Jack,

This is the thread I have been looking for!
I work for an edibles manufacturer in WA state, and I have been charged with the creation of a sample protocol for our large purchased lots of flower material to obtain that accurate "sample representativeness". Based on our GC manufacturer's (SRI) standardized flower sample analytical procedure of 100mg flower in 40mL alcohol, I have expanded their technique to encompass the much larger masses we work with. Briefly;

1) Randomly sample (blind coring of flower lot spread across a table at ~uniform depth) flower lot, acquire >=20g from entire manifest (these can be anywhere from 2-50+ kg flower material).
2) Halve sample => store ~10g for retention, homogenize remaining 10g
3) Sample ~2g from homogenate => spin for 30 min in 400mL denat.
4) Divide final sampled homogenate mass by 10 (to match SRI FED: flowers/ethanol dilution) and inject this mixture into GC, using mass/10 as "sample weight)

I repeat steps 3 and 4 twice more for a final average potency, and this value is used on our purchase contracts.

This has proven effective, and I have quite a bit of data at this point to support that claim.

Before devising this method, I ran an experiment to determine feasibility of larger dilution and optimized FED. It was by no means perfect, but I have that data as well. If you would like to see my data/have any new information on your end regarding optimization of what I call the FED, feel free to contact me.

I will end with a pair of questions:
I have seen the solubility of d9THC (I cannot recall if it was the acid or neutral moiety) somewhere around 50 mM in EtOH, or approximately 15.73 g/L. Do you know which it may be; THCA or THC?
It was listed on another standard manufacturer's website, but at this moment I cannot recall. I would assume THC. If the solubility (50mM in EtOH) is for the neutral species, do you have any information on the solubility of the acid or vice versa?

@Josh Alper: we use 95%+ organic grain alcohol for our extraction. I haven't had the time to determine optimized extraction length, however 15-30 mins is definitely sufficient, assuming you do not reach saturation. With that in mind, if you assume your flowers are <30% THC, you can make an educated guess towards the volume of EtOH necessary for 100% extraction.

@Ahmad; three things: it is almost impossible to quantify the amount of chlorophyll present in a flower sample mixture as it depends on length of extraction, solvent system, moisture content of flowers, etc. however as Jack suggested you can remove most of the chlorophyll from solution with activated carbon. You will lose some cannabinoids in the process (just be sure to retain your cakes). Also, the C(s) is hard to filter out, so I suggest using diatomaceous earth (Celite, Norit, FilterAid) as a pre-filtration treatment. Namely, if you are using a Buchner funnel, slurry pack a 2-3 mm deep bed of celite onto the surface of your filter paper and allow it to run dry prior to filtering off the carbon.

Thanks in advance! And like I said Jack, I have written up the SOPs and have the data if you would like to compare notes.

Eric

28. *Gersh Avery* says:
January 8, 2015 at 11:01 pm

I see that some samples have more solvent than others.

Doesn't that have impact of testing results?

29. *Lisa* says:
January 14, 2015 at 4:53 pm

Hello,
I hope you will get my message.
I'm trying to choose 3 solvents in order to perform the extraction of cannabinoids AND terpenes of cannabis.
I was wondering :
– you don't say anything about ACN which seems to not extract chlorophyll (according to the color). According to the histograms, it appears that it has a good extraction potency. With MeOH leading to high GC maintenance and ethyl acetate which seems to extract chlorophyll relatively well, would you advice working with ACN ? Thaemajor inconvenient that I see is that it is really expensive..
– which leads to my second question. Considering that MeOH shows the best extraction potency, would it be interesting to extract with it, then evaporate to drynesss and then reconstitue in ACN, in which chlorophyll is not soluble ? Or even in hexane…
– do you have an idea about the quality of terpenes extraction with these solvents ?
Thank you !
Lisa

30. *Dustin* says:
January 15, 2015 at 1:12 am

Have you compared MeOH with 10%CHCl3 MeOH?

31. *Jack Cochran* says:
January 17, 2015 at 5:03 pm

Hi Dustin:

No, we have not compared 100% methanol with the solvent mixture chloroform:methanol (10:90). This is a good idea, as I understand this is an industry solvent system that could lead to better extractions of material, but we find it difficult to do these types of experiments as PA is not a medical cannabis State. As such, our experiments have been on illicit marijuana that the police have confiscated, and the experiments are conducted in the presence of the police. Have you compared extraction efficiency of methanol and chloroform:methanol? If so, can you describe your results?

Thanks for reading ChromaBLOGraphy!

JC

32.  *Jack Cochran* says:
January 17, 2015 at 5:15 pm

    Hi Lisa:

    You are describing what I would see as the benefits for acetonitrile very well in your comment. I still think more experiments are necessary, but since PA is not a medical cannabis state, we generally will need to rely on experimentalists in states where cannabis is legal to do that work.

    I don't advise evaporating an extract to dryness and reconstituting as a general practice. Evaporation takes time and equipment and is extra handling, probably more so than is necessary, that will potentially compromise quantification for potency, especially if the reconstitution is inefficient. For example, it is possible that the chlorophyll could hold on to the cannabinoids and the acetonitrile would not be able to redissolve them. Another bad thing about evaporation in this case is that it will almost surely lead to losses of volatile terpenes if you go to dryness (or even if you don't go to complete dryness).

    We don't have good information about extraction of terpenes with all of these solvent systems, since that was not the focus of the work, but Amanda Rigdon has started to do some work with terpenes in hops and I believe will be publishing some of that work later. In the mean time, if you look on ChromaBLOGraphy you can check out her work using the full evaporation technique with Headspace GC-FID. Better yet, we have an application note on the subject at the link below.

    http://www.restek.com/Technical-Resources/Technical-Library/Foods-Flavors-Fragrances/fff_FFAN2045-UNV

    Thanks for reading ChromaBLOGraphy!

    JC

33.  *Jack Cochran* says:
January 17, 2015 at 5:33 pm

    Hi Gersh:

    I am sorry for the confusion, but all samples were extracted with the same volume of solvent, 40 mL (see text in the blog post), even though the photograph doesn't illustrate that. We took extract out of some of the vials for other experiments prior to photography. Next time I will make it more clear and more easily comparable. There is one more thing I should point out, although it is in the text, also, and that is we only had just over 0.1 g of material left for the acetonitrile extraction. Otherwise, the sample amounts were ~0.2g each extracted with 40mL each of solvent.

    You are correct in your comment though: different solvent volumes used could possibly impact quantitative numbers and comparison of results.

    Thanks for reading ChromaBLOGraphy!

    JC

34.  *Jack Cochran* says:
January 17, 2015 at 6:22 pm

    Hi Eric:

Your comments and observations and techniques seem very strong to me. I very much like what you're doing to try and achieve sample representativeness early on. It's certainly a cliche now, but without getting a representative sample, the numbers generated could be garbage, or not fit for the purpose, which seems important given that it's medicine involved.

One thing that would be interesting (unfortunately we can't do it here because PA is not a cannabis state or we would have done it already) is to draw off the solvent completely from your first extraction, re-extract the same cannabis exactly as you did before, analyze that extract, draw off the solvent completely from the second extract and analyze it, re-extract a third time and analyze that extract. Sum the totals for the cannabinoids from the 3 analyses and then calculate the extraction efficiency for each extraction you did. This would be a great start for answering the questions you pose regarding solubility of THCA, since that will be at least one component of extraction efficiency.

You could try backing some of this information out by considering that our stock standards, including THCA, are 1000 µg/mL (1 mg/mL) in methanol, so that is likely a conservative "solubility" (I don't have solubility information for THCA and THC, but I will dig around and see if I can locate it). You could back calculate an extract concentration like seems to be suggested by Josh with that 1 mg/mL value, ignoring for a moment that you guys are using ethanol instead of methanol. Consider that you have 400 mL solvent, so that would be a potential of 400 mg of d9-THCA, let's say. That is 400 mg per 2g, which I believe calculates out to 20% THCA for the original sample. That is surely a very conservative "limit" based on the fact that THCA is probably more soluble in ethanol than methanol, but I think you have to ask yourself how many times you get 20% samples, among other things, when you're thinking about your extraction method.

I would be thrilled to see any results you get from the proposed multiple extraction experiment.

Thanks for reading ChromaBLOGraphy!

Jack

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