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Investigating Lost Spirits Investigations Part II

Posted on **April 25, 2015**

This is a look at the second research paper from Lost Spirits. Eventually I'll take a look at the Wired article that describes their fake aging process. It is really short, barely a paper, and I don't remember people sharing this one around the web, but lets take a look anyhow.

It seems that Lost Spirits got even more analysis equipment and is now exploring GC-MS so the paper is just a demo of their analysis rig up and running. They hope analysis can teach them more about the products out there on the market which they admire so they can attempt to produce similar products. I personally hope to get deeper into analysis but I'd go about it a little differently. They are sort of reinventing the wheel and ignoring a gigantic body of research out there that can be used as a guide. Apparently its easier to buy analysis equipment than to go to the library. This paper cites no references and their last paper only cited one paper from 1908.

The novel approach I've taken to learn more about distillation is to invent small scale concept recipes that illustrate hard to reach concepts in physics and chemistry. These recipes are featured in my distiller's workbook. There is organoleptic analysis using our own organs, where we smell and taste, and then there is analysis like titration, spectroscopy, and chromatography that can objectively count up chemical compounds. My workbook exercises are all based on organoleptic analysis and are designed to sharpen tasting skills by seeing more abstracted nth degree examples. My exercises are also astoundingly more affordable and can teach so much without committing to time on a big rig where it can cost you hundreds per batch to run the still.

Let's jump into Lost Spirit's paper:

> *We learned that the VOC range aroma compounds (primarily fruity esters) mature concentration appears to be predetermined prior to barreling.*

I think this idea here will be a big influence on their accelerated aging technology. The thought is that you can create an new equilibrium with products extracted by oak then use catalysts to

bring the various reactions to the new equilibrium quickly (reaction kinetics). Unfortunately nothing is that simple and other variables come into place if you want a product to be its most extraordinary on a sensory level.

> *We also learned that low rectification products (generally pot distilled) exhibited far more ester precursors and thus the ability to age longer and to a greater aroma and flavor density than the more common high rectification rums.*

Its hard to say anything was really learned. They only looked at one sample. When they say "low rectification products" they mean stuff distilled at a low proof where the congeners are going to be more spread out across the spirits run and a heads cut isn't going to remove as much. More highly rectified rums are typically done on a column still and the proof you distill at implies the congener level. So you're going to make your heads cut governed chiefly by managing ethyl acetate and acetaldehyde then the question becomes: how much other stuff stays in the hearts when your busy focusing on the big two congeners?

> *In this follow up document, we looked at the semi-volatile organics (SVOCs) to see how they change during maturation. The SVOC range includes the majority of maturation compounds including phenolic aldehydes extracted from the oak, medium chained carboxylic esters, complex esters, phenylated esters, higher alcohols, furanic aldehydes, etc.*

So this paper starts to recognize the contribution of other congener categories. All of these compounds are discussed in the abridged bibliography I laid out in Part I. In an article I wrote long ago, from free fatty acids to aromatic esters: esterification in the still make simple(r), I posited the idea that still operation decisions focuses on esters and the other congener classes just tag along. I'm not sure how true that is when you look at the most masterful products, but it might be true for the beginner before they deepen their involvement. In that post, which was widely read, there was so much to learn and research that it ended up being really long. Years later I didn't have time to re-edit things so I just put new ideas and corrections in brackets. What I'm getting at is, as you learn this stuff and share your progress, there are going to be miss steps and you should cut people some slack, be constructive, and we should celebrate any effort because so few people give any. As we redistribute consolidated knowledge, we are all learning together. *A high tide lifts all boats*. Don't be a hipster and not recognize how hard it is to get anywhere with this stuff.

> *While the majority of them were explored in the VOC paper (part 1), two additional esters, ethyl decanoate, and ethyl dodecanoate proved very important in the SVOC range.*

There are lots of esters but these two named are relatively less volatile than others which is why they got labelled semi volatile.

> *Phenolic aldehydes like sinapaldehyde and vanillin are byproducts of the thermal decomposition of lignin in oak. They are responsible for a host of flavors in mature spirits ranging from smoky to vanilla pipe tobacco and*

> *wintergreen. As expected they played a major role in the maturation of spirits. While phenolic aldehydes were expected, the extreme importance of sinapaldehyde in particular was unexpected.*

Its a stretch here to say extreme importance. Finding anything too in depth on aldehydes hasn't been as easy as learning about esters. Probably the best paper explaining the aldehydes is *Origins of Flavour in Whiskies and a Revised Flavour Wheel: a Review*. A unique thing about the paper is the introduction where some of the neuroscience of perceiving these flavor compounds is explained as well as the limitations of just counting chemicals.

> *The above two SVOC chromatograms compare and contrast two heavy pot still rums. The sample on the left is freshly distilled heavy pot still rum (from Lost Spirits Distillery in California). The sample on the right is a Caribbean heavy pot still rum aged for 33 years in an oak barrel.*

This is just a crazy apples to oranges comparison. There are just so many variables that could differ in the starting distillate.

> *The mature rum exhibited a significant increase in the esters ethyl decanoate and ethyl dodecanoate. Both of these esters were present in the freshly distilled rum but in much lower concentrations than those found in mature rum. The mature rum also exhibited a high concentration of sinapaldehyde and acetal which appear to be oak derived.*

The acetals are a unique congener category and are highly aromatic. The origins of flavor in whiskies paper does spend some time explaining them. I think I remember some paper somewhere dealing specifically with ethyl decanoate and ethyl dodecanoate.

> *Perhaps most importantly the mature sample exhibited a large complex mass of "white noise" along the bottom of the chromatogram. This "white noise" represents hundreds of different compounds formed during aging or extracted from the oak. However, the concentrations of the compounds are low and the volatility values are so similar that they merge together into one large unidentifiable mass.*

This is a big limitation of the analysis technique and the subject of a lot of papers is just overcoming noise with countless technique that are truly in PhD territory of sophistication. I had done some playing around with soxhlet extraction and clevenger distillation for botanical analysis for gin production, and though the methods are totally outdated for large products, we need to explore what exactly small producers can practically do with the limited resources they have. Small producers can't yet use the analysis techniques of large producers. There is a gap and if we want to improve the quality of small production spirits, we need to explore it. The work of lost spirits is definitely a stepping stone, but its valuable to figure out what exactly they've done so we don't get lost or derailed when other approaches might be more fruitful.

> *NOTE: The 33 year old sample appears to have been aged with added sugar*

> *in the rum. The large mass in the center is primarily sucrose (table sugar) which could not have been extracted from the barrel. Unfortunately, the sugar obscures some of the data.*

I don't quite understand this claim that the sugar was aged with the rum. Couldn't it have been added after the rum was taken from the barrel? What if added sugar became illegal in years since it was barreled? It would be too risky to add the sugar while the spirits were in the barrel.

> *As a distillery seeking to produce high quality products, a semi-volatile fingerprint was needed to establish a gold standard for quality. Without it, it is not possible to objectively determine when a product has attained maturity or if it is developing the correct signature of a mature spirit in process.*

A gold standard for quality would only come from organoleptic analysis. The only way to tell if a spirit has obtained maturity is to taste it. All objective analysis can tell you, with a lot of systematic experimentation, is how to nudge and sculpt a spirit into the extraordinary. *This* effort produced *that*. And we know this because of *controlled* experiments.

> *Unfortunately, the available chromatogram libraries did not contain semi-volatile fingerprints for aged rums. They only contained fingerprints for malt whisky*

As I mentioned in part I, libraries and models are very important for untangling the readings into something meaningful. Ultimately for the spectroscopy, meaningful readings would help at so many parts of the distilling process but you probably can't even download a model as a shortcut. From what I've read, many models for various purposes will be proprietary to a single production. I'm probably not explaining this the best and so much can be said about applying more advanced analysis to small scale distilleries.

> *This project identified the chemical signature of a mature heavy pot still rum providing the missing baseline data to assess maturity.*

My maturity is not your maturity and we could learn a lot from the wine trade. I think most bourbons have spent too much time in oak and are over mature. Other disagree.

> *In the future this method could be used to compare and uncover counterfeits (immature spirits laced with coloring and flavoring additives) by comparing them to legitimately mature rum. It could also be used to compare subtle differences in products aged with different types of woods or to assess alternative methods and compare them against the signature of a legitimately mature spirit.*

Absolutely, and this is what the industry has been doing for countless decades.

> *The addition of caramelized sucrose to the rum was disappointing. While it may be argued that it is part of the style of these rums, it would have been*

> *beneficial to see the chromatogram without the data obscured by the added sugar.*

Strategic sample preparation, as opposed to direct sample analysis can overcome the bias and its done all the time in various papers.

> *While we can compare this chromatogram to whisky chromatograms, in order to gain an idea of what it would look like without the sugar, we cannot obtain a perfect image that way. We must continue to look for a mature rum that does not contain the added sugar in order to gain an perfect unobscured image of maturity. However, this example does provide the majority of the data needed, especially for the compounds with high peak values.*

This just isn't true. They need to read much more. There are analysis techniques out there that can handle everything they need. They are almost there.

Lost Spirits: Read more, go to the library, read the Bostonapothecary, you're almost there!

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