Integrity **IHEMPAWA** Sustainability

Industrial Hemp Association of WA

Building a hemp industry together in Washington State

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Tacoma, WA

The Honorable Greg Ibach
Under Secretary of Agriculture for Marketing and Regulatory Programs
U.S. Department of Agriculture
1400 Independence Avenue SW
Washington, DC 20250

Re: Second Comments on USDA AMS Interim Final Rule Establishing a Domestic Hemp Production Program; Submitted by the Industrial Hemp Association of WA in conjunction with the Association of Western Hemp Professionals.

I am contacting you again to comment as Bonny Jo Peterson, Executive Director of the Industrial Hemp Association of WA(IHEMPAWA) on behalf of our members. IHEMPAWA is a Washington State Hemp Trade Association started in 2017 and were part of a work group of WSDA representatives, legislators and members who spent a year writing the Washington Hemp Commodity Program legislation signed into law on April 26, 2019. We are also founding members of the newly-formed hemp industry trade group, the Association of Western Hemp Professionals (AWHP). Beyond our official comments, we are in support of comments submitted by Washington State University (WSU) Institute of Biological Chemistry, Washington State Department of Agriculture (WSDA), Oregon Department of Agriculture (ODA), Oregon State University (OSU) Global Hemp Innovation Center and the National Farm Bureau.

The data and comments from *WSU below were based on Washington State hemp trials which were organized and directed by IHEMPAWA and it's members on the farm level. Member farmers in WA volunteered to grow for the WSU trials using their labor and resources with genetics donated by member breeders. We had 5 of 7 trial fields which ranged from 1/4 acre to 10 acres for a total of 20 acres with hundreds of samples taken and tested.

As being among the primary authors of official comments, we cite it as illustration [citation in appendix], the main points of which are listed below:

> *Our proposed adjustments to the USDA Rules require that USDA develop a program which incorporates the following four critical elements:*
>
> 1. *A sampling program based on using a homogenized sample that consists of 25% -30% flower and the remainder being made up of stem/leaf and stalk.*
> 2. *A testing program that is at least 30 days before harvest or a post-harvest test utilizing an HPLC platform for compliance.*
> 3. *The sampling and testing Measurement of Uncertainty (MU) has variables requiring a 30%+ allowance.*
> 4. *Drying is part of harvesting, therefore we need an allowance to move a crop from field to drying facility prior to testing results even if that entails moving the crop between independent license holders.*

The WSU data will show how a 30 day whole plant homogenized sample is representative of the plant in ways the 15 day IFR does not and following what it states with it's short comings is not representative of the plants in the field. A post harvest dried weight whole plant homogenized sample is the truest representation that we can get to with all of the variables at play to show compliance to enter the market with. Post Harvest sampling collection and testing research is being conducted as harvest comes in with data unavailable at time of submission. This research is on 1/4 cup from 1000lb lots and 10,000lb lots of dried material to compare to official state tests in WA and OR. A post harvest test based on a homogenized whole plant sample was passed as part of our new WA hemp program. Under the IFR, we are not able to use the procedure for sampling and testing as planned.

Field sampling and sample prep with so many phenotypes in hemp plants is extremely difficult to do accurately and reproduce with an approximate MU at 30% variability of delta-9 + THCa levels. To get even an approximate representative sample of a field, at least 10 plants per acre are required from the whole plant made up of 20%-30% flower and 70%-80% leaf, stem and stalk. Large grows over say, 500 acres would require more research to determine the number of samples taken. When prepping the wet samples for testing at the lab, drying the samples should be done at 70 degrees F for stability. Filtering out the stems and stalks from the top 1/3 of the plant below the flower is not representative of the plant or plants in the field as an example of lot sizes. Sampling and testing instructions under the IFR Guidelines are incomplete to begin with but we do understand this was a first attempt and revisions would be likely.

After sample analysis of whole plants from one field, it was evident that stalk, stems and flower make up approximately the following percentages after the biomass was divided.
(in % by weight of sample)

Flower: 30%
Medium Stems: 17%
Leaf Stems: 3%
Stalk: 13%
Leaves: 37%

As mentioned previously and by many others, the 15 day pre harvest test is not long enough with testing turnaround time with harvesting and drying issues. Most first year hemp farmers planted late and harvested in late September and will be through October. Multiple hemp samples were delivered on the same day to the WSDA contracted testing labs in 2019 and 2020 causing 5-14 day turn around times where farmers may have had to wait for testing results before harvesting and moving their crops to be dried off the licensed property. With weather issues and uncontrollable circumstances, as the west coast experienced with fires this season, harvesting can require emergency timing or be delayed. The 30 days is not perfect but is possible with proper sampling and testing procedures in place as we learn. The 15 day pre harvest is and will be next to impossible for all farmers. If farmers have to harvest early or late, they can lose yield and income resulting in less tax dollars in state and federal systems ultimately.

Regardless of pre or post harvest compliance testing or timeline, a system with tracking from field to drying facility and to field or storage facility then tested would be inline with all other agricultural commodity testing practices, which all have post harvest testing protocols established. End product use should be taken into consideration.

Without a standard validation protocol, HPLC the current hemp industry gold standard for testing, should be considered as the acceptable USDA testing platform for hemp cannabinoid profiling to determine legal THC crop results. Gas Chromatography uses high heat to analyze industrial hemp samples. This platform is the gold standard for testing terpenes and residual solvents not cannabinoid profiles with ease and accuracy. Potencies can be done on a GC/MS, but the high heat decarboxylates the acidic forms of the cannabinoids and therefore the data yielded are only the active forms of the cannabinoids (e.g. THC and CBD). To get a more accurate potency you would need to apply a derivatization calculation to derive the acidic forms of the cannabinoids. HPLC involves separating cannabinoids through a column matrix. This method preserves the acidic forms of the cannabinoids (e.g. THC-A and CBD-A).

Washington is under our new program as of January 1, 2020 and we do not have the option to continue under the 2014 IHRP due to state statue. Expedited changes to sampling and testing before the 2021 season begins are crucial to our hemp industry in Washington and nationally.

Thank you for your time and consideration,

Bonny Jo Peterson,
Executive Director
Industrial Hemp Association of WA
(for the membership of the Industrial Hemp Association of WA)

Board Members

Dylan Summers, Lazarus Naturals
Bill Cyr, Unique Food Works
Robert Cook, Columbia Valley Hemp Co.
Kristofer Plunkett, Cascadia Hemp Co.

Attachments/Sites:

***Washington State University Comments and Data "Evaluation of methods used to sample hemp for regulatory compliance testing" [attached in appendix I]**

Association of Western Hemp Professionals [attached in appendix II]

https://www.perkinelmer.com/CMSResources/Images/44-174745APP_Cannabis-Analysis-Potency-Testing-Identifification-and-Quantification-011841B_01.pdf

Identification and quantification of cannabinoids in Cannabis
sativa L. plants by high performance liquid
chromatography-mass spectrometry
Oier Aizpurua-Olaizola & Jone Omar & Patricia Navarro &
Maitane Olivares & Nestor Etxebarria & Aresatz Usobiaga,
Received: 24 July 2014 /Revised: 4 September 2014 /Accepted: 9 September 2014 /Published online: 23
October 2014, Springer-Verlag Berlin Heidelberg 201

Appendix I

Washington State University (WSU) Data

Evaluation of methods used to sample hemp for regulatory compliance testing

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As outlined in the “Establishment of a Domestic Hemp Production Program; Comment Period Reopened” document, multiple comments made during the previous open comment period for the Interim Final Rule (IFR) raised clear questions regarding the “uncertainty of sampling” of hemp samples being analyzed to determine field compliance with legal THC levels for hemp (being defined as ≤0.30% THC by dry weight).

In order to address this concern and to better understand how best to sample hemp plants to test for total THC (delta-9-tetrahydrocannabinol) potency compliance, researchers at Washington State University (led by Professor David R. Gang, Fellow in the Institute of Biological Chemistry and the Assistant Director of WSU’s Agricultural Research Center and the CAHNRS Office of Research in the College of Agricultural, Human and Natural Resource Sciences) were asked by the Washington State Department of Agriculture (which oversees Washington State’s Industrial Hemp program) to compare the new sampling protocol that was implemented by the USDA for 2020 to the method approved for use in Washington State in 2019.

These researchers performed a series of experiments to compare the sampling methods to each other (and to alternative methods) with the aim of determining how consistent (or not) the methods are relative to each other in being able to accurately and reliably determine total THC levels within hemp plants in the field.

Total THC levels are determined by adding free THC levels and THCA levels (Tetrahydrocannanolic acid, corrected for the difference in mass of the two compounds) using the formula: Total THC = THC + THCA*0.877, per the IFR. This formula takes into account the difference in mass of THC (formula $C_{21}H_{30}O_2$, 314.469 $g{\cdot}mol^{-1}$) and THCA (formula $C_{22}H_{30}O_4$, 358.478 $g{\cdot}mol^{-1}$); with 314.469/358.478 = 0.877234. Rounding down the multiplication factor to 0.877 leads to a very slight (0.0266%) underestimation of the total THC contribution by THCA in the calculation. This difference is well below anything significant compared to the chemical level variability in the hemp plants being analyzed or the accuracy of high-end, state of the art analytical instruments available today, as outlined below. The calculations used in this analysis do not take into account questions raised in the original comments related to the “Liquid Chromatography Factor, 0.877” regarding potential loss of THCA to thermal degradation instead of being converted at a 100% efficiency to THC. This report therefore recognizes that levels of actual “total THC” that is physiologically relevant could be lower than determined using the calculation-based methods employed herein. That fact in no way changes the conclusions of this report with regards to the adequacy, reproducibility and robustness of different sampling methods to be employed when determining THC levels in hemp fields.

Four different sampling methods to be compared.

2020 Top 1/3 Plant Sampling Method: This is the current USDA-approved sampling method employed in 2020, with each field sampled at 15 days preharvest, and the top 1/3 of representative plants in the field being used for analysis. Briefly, "representative field samples" are collected and then pooled to form a combined sample per field, which is then dried. A "representative" subfraction of the dried plant material is ground either to a dry powder or directly in a solvent (depending on the specific implementation in the performing laboratory), and is then extracted by a solvent (typically methanol) and then analyzed by a method (either gas chromatography- or liquid chromatography-based) that measures total THC levels (either post-decarboxylation or combining data from free THC and THCA) to determine the "total THC" level for the field. During the implementation of this sampling process, a number of plants are sampled per hemp field, wherein a single branch from the top 1/3 of each plant is collected and added to a pool of branches per field, with the number of plants sampled being determined by the size of the field. The larger the field, the larger the number of branches sampled, but a minimum of 10 branches are typically sampled. These branches are then dried at a temperature low enough to efficiently remove moisture while maintaining THCA integrity. It is well-known that heating THCA to temperatures in excess of 40 °C (104 °F) leads to decarboxylation of THCA to form free THC. It is for that reason that "total THC" levels are measured and not just free THC, because processes such as heating (as occurs in smoking) converts THCA to THC, thereby leading to potential psychoactive compound levels that would exceed the legal limit as defined for hemp in the 2018 Farm Bill. Once the branches are dried, the dried flower and leaf tissues are removed from each branch and representative sample is taken from the dried plant material per branch (including leaf, flower and stem), then mixed into a large pooled sample containing dried plant material from each branch. From this pooled sample, a representative sample is taken and extracted and then analyzed for cannabinoid profile, with Total THC levels being determined using the formula shown above. It should be clear from the above written description, that the word "representative" is used multiple times in the description of how to perform the sampling and then extraction of those samples to generate a number used to determine if a particular hemp field is compliant or not regarding THC levels. A "representative" tissue sample from "representative" branches from the top 1/3 of "representative" plants is what gets extracted.

The way this procedure is typically implemented, just 0.2 g of plant material is actually extracted for the compliance analysis per hemp field. That is a very small amount of tissue relative to a large field, and leaves open the issue of potential stochastic sampling error. If the field contains plants that are not completely uniform in their THC levels relative to each other, it is possible that this small subsample in any given analysis could over-represent plants that have higher levels of THC, thereby leading to failure of the field. On the other hand, equally possible, that analysis could over-represent plants that have lower levels of THC, leading to passing the field. This issue is addressed below in more detail. Indeed, as the results presented below clearly demonstrate, we found that such sampling errors are not only possible, but the likely result in any given test. In other words, the most likely result of a sampling test is an inaccurate assessment of the total THC levels based on the method used to sample the plants in the field and then prepare them for extraction.

After extraction, as indicated in the comments provided in the previous open comment period, the results are very consistent. Indeed, the method used to measure the total THC levels in the extracts was found to be extremely reproducible in the hands of the WSU researchers. During the analyses performed at WSU, the researchers included a series of quality control (QC) samples, all being aliquots of the same exact mixture of cannabinoid standards and run periodically throughout the analyses that were performed over many days. Despite the fact that these QC samples were analyzed on different days, the WSU researchers found that the coefficient of variance of the results for THC levels in those QC samples was less than 1% (0.581% in fact). Clearly, the methods for measuring THC in extracts are highly robust and reproducible. The question then arises regarding the same for the sampling procedure, as described in more detail below.

2019 Whole Plant Remediated Sampling Method: This method was employed in Washington State in 2019 (WSDA approved method) and collected representative "whole plant remediated" samples at 30 days preharvest, which included taking leaf, flower, and stem samples from regions of the whole plant (bottom 1/3, middle 1/3, top 1/3). In this sampling method, the leaf and flower tissues are dried, crushed separately and a "representative sample" of the dried plant material is then ground separately by tissue type and fractions thereof taken at a ratio of 37:30 and combined, or the tissues are mixed at that ratio prior to grinding. The ground sample is then extracted and analyzed as for the 2020 Top 1/3 Plant Sampling Method described above. This ratio (37:30) represents the fraction of the hemp plant at 30 days to 15 days preharvest that is leaf tissue or flower tissue, respectively, with the remaining 33% of the tissue of a hemp plant consisting of stem/stalk tissue. Because only flower and leaf material is extracted using this method, the sample is "remediated" by

multiplying the determined total THC level by 0.67, thereby taking into account the mass of the plant made up by stem/stalk. The same issues described for the 2020 Top 1/3 Plant Sampling Method regarding potential stochastic sampling bias due to small amount of tissue per field being actually extracted holds for this sampling method. Again, a "representative" tissue sample from "representative" plants is what gets extracted. Again, that representative tissue sample is a very small sample size in actual fact (~0.2 – 0.25 g as typically implemented).

2020 Whole Plant Remediated Sampling Method: This method follows the procedure for the 2019 Top 1/3 Plant Sampling Method, but collects the plant material to be analyzed at 15 days preharvest instead of at 30 days preharvest. The reason for including this sampling procedure was to enable robust comparison of the sampling methods, to eliminate any potential bias that could be introduced by sampling plants 2 weeks apart and then comparing their THC levels.

2020 Top 1/3 Plant Individual Plant Sampling Method: This method was implemented by the WSU researchers to determine what level of cannabinoid variability actually existed in the fields that were used for the analysis described in this report. Plant branches were collected and dried as described above for the 2020 Top 1/3 Plant Sampling Method. Indeed, for the analysis performed by WSU researchers in 2020, the same exact branches were used for the individual plant analysis that were used following the standard USDA method described above. However, prior to mixing the dried plant material into a large pooled sample, individual samples per plant (half of each branch) were separated out to be analyzed separately by plant branch, thereby enabling the analysis and comparison of individual plants and determination of the variation of cannabinoid levels present in the field. In total at least 10 plants per field were thus individually sampled and THC (and other cannabinoid) levels measured.

Implementation of Sampling Procedure in Washington State in 2020.

The WSU researchers collected all field samples required to perform the analysis described in this report. Indeed, the same individual, Professor David Gang, performed all sampling in the field. And a single researcher (a senior Research Associate in the Institute of Biological Chemistry at WSU) performed all extractions and chemical analyses in the lab. The reason for involving just these two individuals in the analysis at WSU was to ensure that no investigator bias would be introduced in the analysis.

Samples were collected from 5 separate fields in the eastern half of Washington State, designated in this report by the name of the town that is closest in proximity to the field sites. All were outdoor grows, with plants grown in soil in the field using different cultivation methods (different fertilization regimes, different weed control measures, different plant spacing, etc.). Different hemp varieties were used in these fields, either as single varieties per field or mixed varieties per field, so that results could be extrapolated to other hemp varieties in Washington State, in other words so that robust conclusions regarding the analysis could be drawn that would apply to hemp in general, and not just a single hemp "variety". Herein "variety" means a breeding or cultivar stock that is grown in the field for purposes of hemp production either for research purposes or for ultimate product generation.

Samples for THC potency compliance testing by the WSU researchers were collected from each of those 5 fields on 2 separate occasions (30 days preharvest collection on August 24, 2020 and 15 days preharvest collection on September 7, 2020 [except for the samples collected for 15 day preharvest at the Elk field, which were collected on September 8, 2020) per each sampling protocol as described above, with at least 10 plants per field sampled for the analysis. Samples were placed in the field into labeled paper bags, kept in a large cooler to keep the samples cool and then transported by SUV to a laboratory in the Institute of Biological Chemistry on the Pullman campus of Washington State University, where they were laid out on screens to be dried at <24 °C (<75 °F) in a room with low relative humidity and a light fan breeze blowing over the samples, to prevent any mold formation or rotting of the plant material while drying. Once the samples were dried, they were separated out by tissue type as indicated in the method procedures and partitioned into representative samples to be processed either at WSU or at other labs. Dried samples for each field were thus processed at WSU using the standard extraction procedure implemented in Washington State, and an aliquot of each sample was sent to a separate research lab within Washington State. With 5 fields included in the analysis and 4 separate sampling methods employed, a total of 200 individual samples were dried, ground and analyzed for total THC levels in this method comparison test. These samples were each analyzed by two labs (WSU, second research lab in Washington State) and results of those analyses compared.

Results from Comparison of Sampling Methods

As can be seen in the following tables and charts, the sampling method employed can have a large impact on the results obtained. Note: as described above, the method of analysis after extraction that was used by the WSU researchers and the second research laboratory is very robust and reproducible, with the coefficient of variance (% error) of the technical, analytical method being less than 1% (just 0.581% for WSU) in these analyses. What was observed in the hemp analysis data (see Table 1), on the other hand, was a relative percent error in the analysis that was typically an order of magnitude higher, ranging from 3.2% to 13.8% for the remediated sampling methods to as high as 54% to 67% for the top 1/3 plant based methods. This large variation in results can only be ascribed to the lack of ability of the sampling procedure to generate a consistent, reproducible sample from any given hemp field. Because this error was observed over and over across multiple hemp fields growing different hemp varieties/lines, it is a systemic problem with the sampling procedure and not an artifact of analysis of one particular hemp line.

Table 1. Comparison of Sampling Methods for Total TCH Determination

Total THC (% in sample), Average of 10 samples measured					
Field	**30d remediated**	**15d remediated**	**15d top1/3**	**15d top1/3_individuals**	**15d top1/3_HS**
Prosser	0.087	0.071	0.176	0.157	0.220
Elk	0.038	0.106	0.343	0.071	0.070
Kennewick-CB	0.067	0.237	0.452	0.435	0.200
Kennewick-YB	0.125	0.187	0.391	0.241	0.240
Mabton	0.027	0.039	0.066	0.058	0.070
Standard Deviations (% in sample)					
Field	**30d remediated**	**15d remediated**	**15d top1/3**	**15d top1/3_individuals**	**15d top1/3_HS**
Prosser	0.020	0.012	0.186	0.296	0.450
Elk	0.004	0.032	0.213	0.024	0.030
Kennewick-CB	0.014	0.056	0.190	0.766	0.410
Kennewick-YB	0.023	0.081	0.422	0.416	0.510
Mabton	0.003	0.006	0.031	0.048	0.040
Standard Error (% in sample)					
Field	**30d remediated**	**15d remediated**	**15d top1/3**	**15d top1/3_individuals**	**15d top1/3_HS**
Prosser	0.006	0.004	0.059	0.094	0.142
Elk	0.001	0.010	0.067	0.008	0.009
Kennewick-CB	0.005	0.018	0.060	0.242	0.130
Kennewick-YB	0.007	0.026	0.133	0.132	0.161
Mabton	0.001	0.002	0.010	0.015	0.013
Relative Percent Error (%)					
Field	**30d remediated**	**15d remediated**	**15d top1/3**	**15d top1/3_individuals**	**15d top1/3_HS**
Prosser	7.2	5.3	33.4	59.6	64.7
Elk	3.2	9.6	19.6	10.7	13.6
Kennewick-CB	6.8	7.5	13.3	55.6	64.8
Kennewick-YB	5.9	13.8	34.1	54.5	67.2
Mabton	3.3	4.7	14.7	26.1	18.1

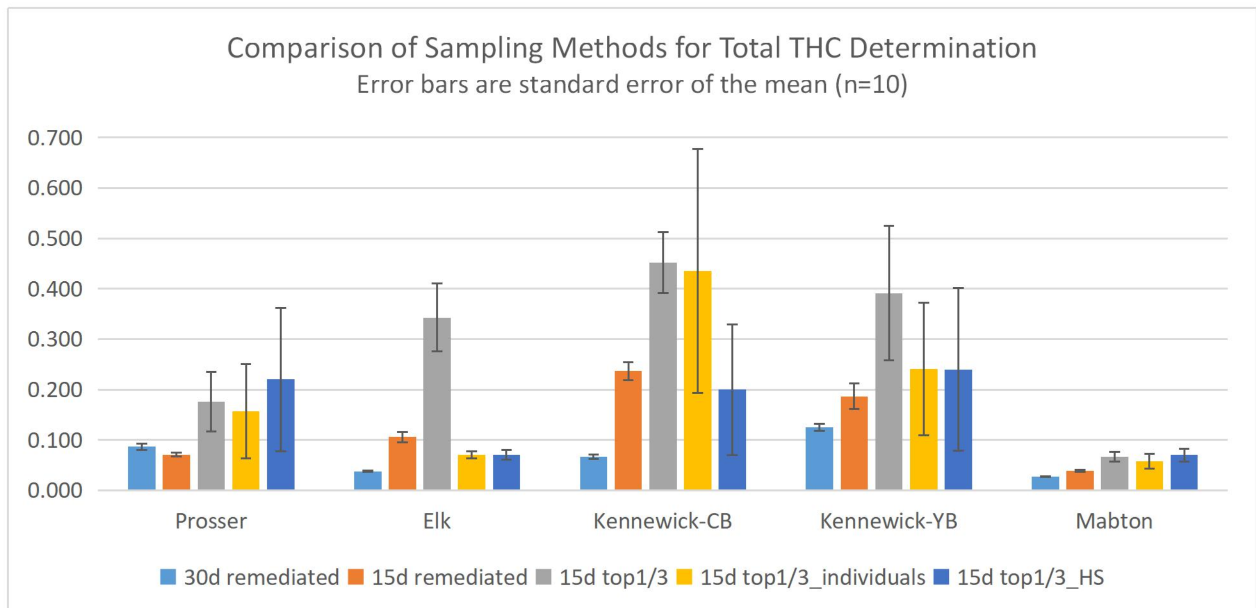

Figure 1. Comparison of total THC levels determined for five different hemp fields when sampled using four different sampling methods and then analyzed at two different analytical laboratories. The 30 day preharvest remediated whole plant sample (30d remediated), the 15 day preharvest remediated whole plant sample (30d remediated), the 15 day top 1/3 plant representative sample (15d top1/3) and the 15 day top 1/3 plant individual plant sample (15d top1/3_individuals) samples were all analyzed in the Institute of Biological Chemistry at Washington State University. A second analysis of the 15 day top 1/3 plant individual plant sample (15d top1/3_HS) samples was performed at a separate research lab in Washington State.

Indeed, when data for individual plants used in this analysis were evaluated closely, it became apparent that THC levels varied significantly from plant to plant, and this was most pronounced in the top 1/3 of the plant samples. For example, in the Kennewick-CB field, THC levels ranged from 0.06% to 2.46% in the top 1/3 plant samples when individual plants were evaluated separately (more than half of the plants had THC levels less than 0.08%, only four exceeded 0.1%, with three of those being "hot" and exceeding 0.3%). In the pooled sample for the 15 day preharvest top 1/3 plant analysis, the THC levels ranged from 0.24% to 0.85%. And, in the whole plant remediated samples for this field, also collected at 15 days preharvest, these levels ranged from 0.16% to 0.31%, with only one sample exceeding 0.3%. If all plants in the field had approximately the same levels of THC, the measured values would have been close to identical based on the robustness of the analytical procedure. If the variation in plant THC levels were to be uniformly distributed across the field, it again would be expected that, on average, the values obtained from a uniform sampling procedure would generate an accurate measure of the field-level THC levels. However, the sampling experiments that were performed indicated that such uniform distribution does not occur at the field level, at least for the hemp lines that were grown in the fields evaluated here. Individual plants with significantly different THC levels than the average for the field can have a large impact on the results obtained for the field. If even a single plant has high THC levels, and that plant happens to be included in the analysis, it can significantly skew the results, especially, apparently, when the top 1/3 plant sampling method is used.

It is obvious from these results that the current USDA method, which analyzes only the top 1/3 of the plant, generates data that is very error-prone and results that likely do not represent the actual THC levels that are present in the hemp plants in the field as a whole. Following the current method, it is thereby possible, indeed likely, that stochastic effects come into play in the analysis and can significantly affect the outcome. An alternative method, such as using a whole plant remediated sample or testing samples post-harvest is warranted.

Ring test to determine lab-to-lab variability

A ring test experiment was performed to determine what type of variation in analysis could be expected if different analytical labs were to test a given sample. Samples for this experiment were generated using the official 2020 sampling method (2020 Top 1/3 Plant Sampling Method) as described above and sent to analytical labs as would typically be done by the WSDA for a field being compliance tested. Three separate commercial labs that are approved to test for the WSDA were included (they were not told that they were involved in this experiment, samples were just sent to them for analysis as any grower would send them, so

that their results would not be biased by a desire to compare favorably to other labs). Thus, it was a blind experiment with regards to the commercial labs. In addition, the WSU lab and the other research lab in Washington State that was involved in the other experiments described above also tested the same samples. These samples consisted of plant material collected from the top 1/3 of the plant from at least 10 plants in 5 separate fields (following the standard approved procedure, as described above). The aggregate/pooled sample per field of crushed hemp material was then mixed well and a sub-sample (4 – 5 g of dried plant material consisting of mostly leaf and flower tissue, but also some small stems, as is typically collected for the top 1/3 of the plant sample type) was then sent to each lab for analysis. Thus, a total of 4 measurements (4 labs) were made on samples from 5 separate fields. Results of this analysis are presented in the graphs below.

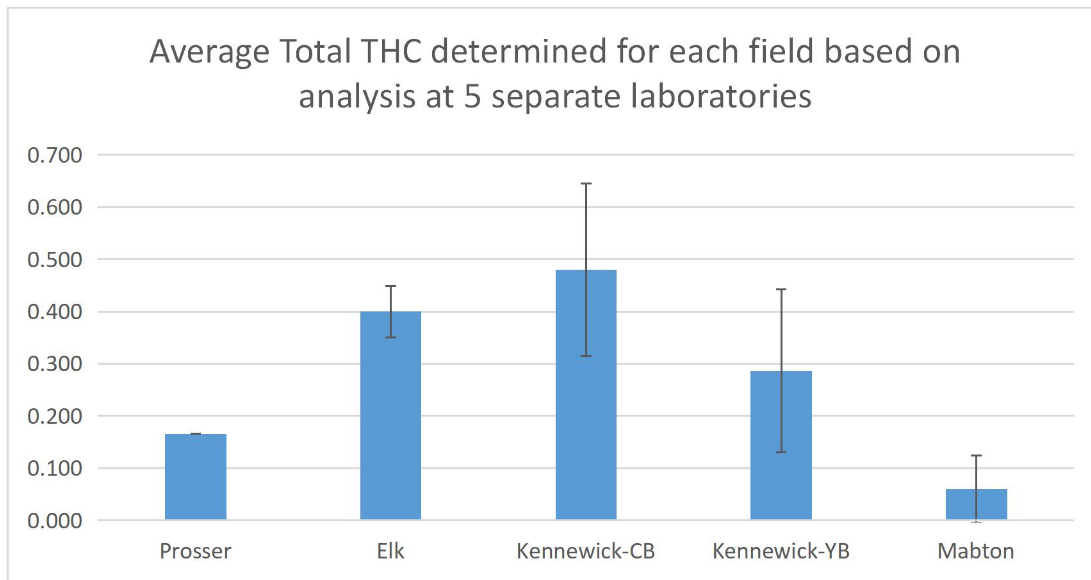

Figure 2. Average Total THC determined for each field included in this analysis, based on analysis performed at 5 separate analytical laboratories. The error bars are standard deviations and help show the variation in the results obtained.

It is important to note that on average, as shown in Figure 2, all fields except Kennewick-CB and Elk would pass compliance if the average of these four labs were taken. What was surprising was that the Elk field would not pass on average, even though all data from the analysis described above comparing the sampling methods clearly showed that the Elk field had, on average, a low total THC level, when alternative methods of sampling were employed. It was the nature of the top 1/3 plant representative sample method, which is very prone to stochastic effects (one small piece of "hot" tissue in the sample that is taken can significantly impact the result observed), that led to this result in the ring test. When the Elk results were evaluated at the individual plant and individual sample level, it was found that the overall THC levels are low, but a few plants have elevated levels. Over-sampling of the "hot" plant tissue can therefore lead to a non-compliant test result even if the field on average is compliant. The standard deviation in this comparison was relatively high (ranging from 22% – 41%, depending on the field), again indicating that the specific sub-sample that is ultimately selected for extraction and analysis plays a large role in THC level determinations when using the current sampling method.

Table 2. Average Total THC determined for each field based on analysis at 5 separate laboratories

Field	Total THC (%)	Stdev	Percent Deviation
Prosser	0.166	0.049	30%
Elk	0.400	0.165	41%
Kennewick-CB	0.480	0.156	33%
Kennewick-YB	0.286	0.064	22%
Mabton	0.059	0.020	34%

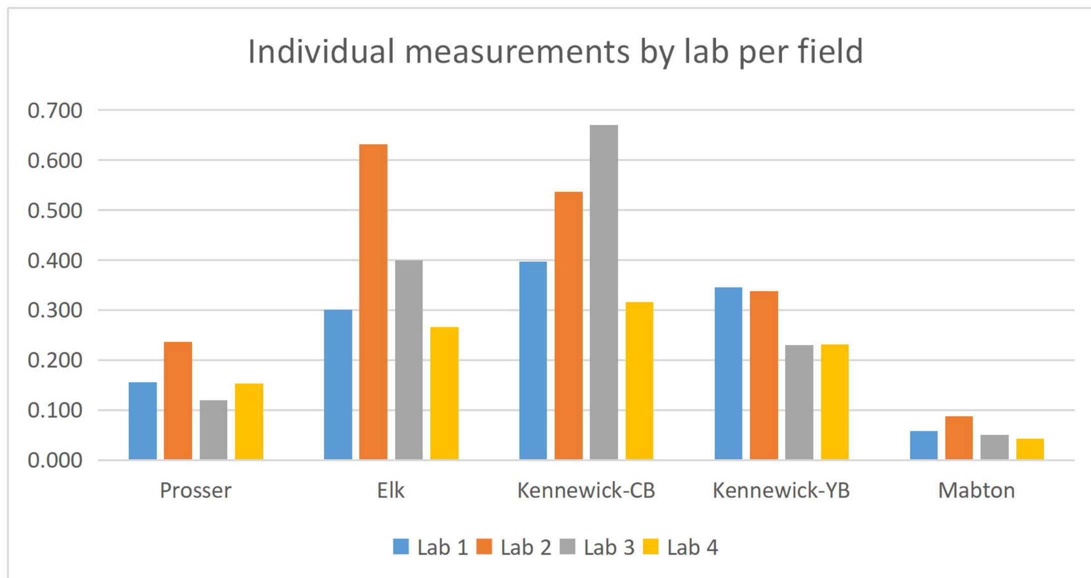

Figure 3. Individual results per lab for compliance testing of the same sample (crushed hemp tissue, not powdered and homogenized).

Overall Conclusions

As described above, significant variation should be expected when analyzing samples collected using the 2020 Top 1/3 Plant Sampling Method (current official USDA method). That was indeed the case for the SAME sample sent to each of 4 labs for analysis. Results obtained back varied by more than 2-fold depending on which laboratory results are compared to each other. This difference was meaningful because the differences led to samples being determined as non-compliant by one lab but compliant by another. Because the sample sent to each lab was from the same pooled sample, and consisted of plant material that was crushed to less than ¼ inch on average pieces but not ground to a fine powder (that grinding was performed in each lab), the exact sub-sample that was ground in each lab was not the same and therefore small pieces of high THC flower material from a "hot" plant can skew the results obtained for an entire field.

The results of this analysis clearly demonstrate that the current sampling method is inadequate and unable to generate reproducible and robust results, either within a single laboratory and especially across multiple laboratories. Alternative sampling methods (not the top 1/3 plant representative sample method) need to be employed to more accurately determine total THC levels in hemp fields grown for commerce. The whole plant remediated method more accurately reflected what was observed when multiple individual plants were each analyzed separately and then the results from their analysis combined into an average value.

Due to the deadline imposed for commenting, it was impossible to compare post-harvest sample results to the 15 day preharvest or the 30 day preharvest sampling results. Harvesting of hemp in WA state is just happening now, immediately prior to the commenting deadline, and compliance testing results take at least a week to be generated if the testing lab has nothing else to do (which is never the case). The question regarding measurement uncertainty (MU) is still open regarding the current sampling method and needs to be revisited.

Comparison of Sampling Methods for THC Potency Testing of Industrial Hemp
2020 vs. 2019 Sampling Protocols

We have collected samples from 5 fields across central and eastern Washington State to aid in our comparison of sampling procedures for potency testing of hemp:

1. Kennewick field 1 = "Yong Bern"
2. Kennewick field 2 = "Cherry Bern"
3. Prosser field = combined "Yong Bern" (80%) and "Cherry Bern" (20%)
4. Mabton field = Tricrop
5. Elk field = combined "Yong Bern" (70%) and "Cherry Bern" (30%)

We have collected 3 separate sample types from each of these fields:

- 30 day preharvest “whole plant remediated sample” (collected 20200824) – 2019 sampling protocol
- 15 day preharvest “whole plant remediated sample” (collected 20200907 – except for Elk field on 20200908) – 2019 sampling protocol, but at 15 days preharvest instead of 30 days
- 15 day preharvest “top 1/3 of plant representative sample” (collected 20200907 – except for Elk field on 20200908) – 2020 sampling protocol at 15 days preharvest

For each of these sample types, at least 10 plants per field were sampled (typically 12 – 13 to insure that at least 10 plants will be in representative samples if some of the plant tissue becomes “corrupted” (e.g., rots, gets crushed and is no longer useable) prior to drying – we didn’t see this problem happen, however). At least 500 g of fresh plant tissue was collected per field using sharp garden clippers, and placed into large thick paper bags (taller and thicker than typical grocery store shopping bags), with each bag labeled with field location, plant genetics if required, and date of collection, and then each bag was placed into a cooler in the collection vehicle to keep from getting too hot on the collection days.

Samples were taken to WSU’s Pullman campus, to the Tissue Imaging, Metabolomics and Proteomics Laboratory (TIMPL), for sample drying and then sample processing and downstream chemical analysis both at TIMPL and at Hops Extract Corporation of America (Hopsteiner) in Yakima.

Processing and drying of whole plant remediated samples (2019 method):

Samples were collected in the field into large paper bags as outlined above, with pieces of at least 10 plants collected such that stems of various sizes, older large leaves, young small leaves, flowers, etc., are all collected and placed into the bag. Due to the nature of the sampling, it was not possible to connect any given piece of tissue to a particular plant. Thus, this method is useful to determine the levels of cannabinoids across a field and to show levels of overall variability, but is not able to determine how that variability is connected to specific individual plants.

Samples of this type were processed such that while the tissues were still fresh, the leaves and flowers were removed/plucked from the stems and all tissues (flower, leaves, stems) were placed into three respective piles on large mesh screens (Home Depot purchased window screen replacements with frames, extended out to 18 × ~30 inches). One screen per field to prevent any chance of cross mixing of samples between fields. The tissues in each pile were spread out and “fluffed” to support efficient air flow around all pieces of the tissues to support efficient drying while also preventing mixing between samples. The screens holding the samples were placed on benches and lab carts in a laboratory room such that air could readily flow around them with a fan gently blowing in the room to circulate the air. The tissues being dried were kept between 22 °C and 24 °C (71 °F to 74 °F) for several days to insure complete drying while also keeping the temperature low enough to be confident that no heat conversion of THCa to THC would occur. The relative humidity level in Pullman in the summer is typically lower than 35%, usually in the 20% to 30% range, which is highly conducive to rapid drying of plant tissues. There was no evidence of any molding of samples occurring under these conditions. Each of the three tissue piles per field was then separated into two equal sized samples, one to be retained at WSU TIMPL for analysis and the other to be sent to Hops Extract Corporation of America (Hopsteiner) for analysis. So, for each field for each sampling day (30 day preharvest and 15 day preharvest), there were 6 separate samples bagged – 2× with flower tissue, 2× with leaf tissue and 2× with stem tissue. Total of 12 bags for the two days.

Prior to extraction for cannabinoid compositional analysis (potency testing) the leaf:flower samples are mixed at a ratio of 37:30, respectively, by mass, crushed lightly by hand and mixed, then separated into 10 separate samples for each field, with each sample constituted such that it appears to be “representative”, i.e., having approximately the same amount of leaf and flower tissue as the other samples. Each separate sample is thus a biological replicate as it contains leaf and flower material that has been slightly crushed to support easy separation into 10 samples, but has not been homogenized and thus turned into one single pooled sample. Each of these 10 samples is then ground to a powder and extracted using the standard approved method utilizing methanol as extraction solvent followed by filtration to remove tissue particles and subsequent analysis by HPLC over C18 column with UV detection.

In the case of samples analyzed at WSU, the HPLC used was a Waters ACQUITY UPLC with inline PDA detector for UV analysis, with post-PDA analysis by MS also performed to provide for extended cannabinoid analysis.

Each sample contains the following tissues at approx. representative ratios for the field at time of collection:

- Flower (30% of mass)
- Young and old leaf (37% of mass)
- Medium stems and leaf stems (20% of mass)

- Old stem/stalk (13% of mass)

Only the flower and leaf are used for chemical analysis, so the stem mass (33%) is then used to adjust the values obtained for THC and other cannabinoid levels to determine a final, by total plant composition (each final value is divided by 0.67 to obtain the final value determined for the cannabinoid).

Processing and drying of top 1/3 plant samples (2020 method):

The top 1/3 plant samples will be analyzed in two ways. One method follows the WSDA IFR 2020 protocol, which generates a "representative sample" for the field. The second method will allow us to evaluate actual variability within the plants across the fields (data we want but is not needed by the USDA). We can do both as outlined below.

Samples were collected in the field into large paper bags as outlined above, with whole branches of plants collected from the top 1/3 of individual plants being placed into a common bag. Two branches per plant were collected so that we had enough to provide samples for the two labs involved in the analysis (TIMPL and Hopsteiner). At least 11 plants were sampled per field (typically 12 – 13) to insure that enough branches were available with sufficient leaf and flower tissue each for the downstream analysis. This method is useful to determine the levels of cannabinoids across a field and to show levels of overall variability, and is also able to determine how that variability is connected to specific individual plants because the flower/leaf tissues to be evaluated in a single sample can come from a single stem. Two types of samples are generated from these collected tissues as outlined below.

Samples of this type were processed such that while the tissues were still fresh the whole, intact stems were placed carefully on large mesh screens (Home Depot purchased window screen replacements with frames, extended out to 18 × ~30 inches). One screen per field to prevent any chance of cross mixing of samples between fields. The branches on each screen were spread out and "fluffed" to support efficient air flow around all pieces of the tissues to support efficient drying. The screens holding the samples were placed on benches and lab carts in a laboratory room such that air could readily flow around them with a fan gently blowing in the room to circulate the air. The tissues being dried were kept between 22 °C and 24 °C (71 °F to 74 °F) for several days to insure complete drying while also keeping the temperature low enough to be confident that no heat conversion of THCa to THC would occur. The relative humidity level in Pullman in the summer is typically lower than 35%, usually in the 20% to 30% range, which is highly conducive to rapid drying of plant tissues. There was no evidence of any molding of samples occurring under these conditions.

The branches from each field, after being dried, are individually placed into Ziploc bags and labeled such that each plant's tissues would be kept separate from the other plants. A total of 10+ samples were retained at WSU TIMPL for analysis and another 10+ samples per field were sent to Hops Extract Corporation of America (Hopsteiner) for analysis. So, for each field for the 15 day preharvest sampling day, there were 10+ separate samples bagged.

Each individual sample (per plant in each field) is then processed such that the leaves and flowers from each stem are stripped off the stem and combined into a single sample (also containing small "stem pieces" (which are typically the petioles). This sample is then partially crushed to mix the tissue well and divided into two sub-samples per plant. One of these samples is used for extraction and individual plant cannabinoid profiling as outlined below. The second sample is combined with the similar samples from the other 9 individual samples per field into one large sample, which is then mixed and sub-divided into 10 samples, where each sample is a "whole field representative sample" of the field. Those 10 samples are each crushed completely, ground, and then extracted as outlined below to be used for cannabinoid potency testing. Thus, these are rather 10 "biological" replicates per field.

We thus have 20 samples to analyze per field for the top 1/3 plant collected sample type. Ten of those are used for potency testing per 2020 IFR method, to compare against the 2019 method, and the other 10 are used for evaluation of plant to plant variability within the fields and overall variability of the method in general.

Prior to extraction for cannabinoid compositional analysis (potency testing) of either sub-sample type for the top 1/3 plant sample type, the total combined leaf and flower material of each individual sub-sample is ground to a powder. For the whole field representative samples, per the IFR, each is passed through a 1.5×1.5 mm mesh screen to remove any non-powdered stem tissues and then used for the analysis. For the individual plant samples, we will not screen them. Each of the 10 samples per sample type is then extracted using the standard approved method utilizing methanol as extraction solvent followed by filtration to remove tissue particles and subsequent analysis by HPLC over C18 column with UV detection.

In the case of samples analyzed at WSU, the HPLC used was a Waters ACQUITY UPLC with inline PDA detector for UV analysis, with post-PDA analysis by MS also performed to provide for extended cannabinoid analysis.

Appendix II

October 8, 2020
Mr. Bruce Summers
Administrator, Agriculture Marketing Service
U.S. Department of Agriculture
1400 Independence Avenue, SW
Washington, DC 20250

Re: Comments on USDA AMS Interim Final Rule Establishment of a Domestic Hemp Production Program; Submitted by the Association of Western Hemp Professionals
Docket ID: AMS-SC-19-0042-4666

Comments submitted via the Federal eRulemaking Portal at www.regulations.gov.

Dear Administrator Summers,

On behalf of the Association of Western Hemp Professionals, a non-profit trade association of small and medium hemp producing, harvesting, extracting and ancillary hemp-derived product
businesses located in the Western states, established for the purpose of lobbying hemp legislation and advocating for the hemp industry from soil to end-user (westernhemppros.org), we appreciate this opportunity to comment on the above-captioned matter.

AWHP filed comments previously on the IFR, which established a domestic hemp production program pursuant to the Agriculture Improvement Act of 2018. We stand by our previous comments while taking this opportunity to add to those comments as allowed by the extension of the comment deadline to October 8, 2020. Reopening the comment period, gives interested persons a needed additional opportunity to comment on the IFR, although in many states we are in the middle of the hemp harvest season--which does limit the amount of quality comments based on the 2020 hemp growing season with many crops still in the field. State Departments of Agriculture, universities and farmers are forced to scramble to compile and comment on their data that was able to be collected by the deadline.

I. IFR Areas of Concern

1: 15-Day Harvest Window

AMS received a significant number of comments on the 15-day requirement during the initial comment period including those from AWHP. Ideally, a post harvest, homogenized, whole plant analysis would be most prudent in determining compliance for market-bound hemp material. At minimum, we support a 30-day period for pre harvest testing of the whole plant, homogenized and cite to recent findings by Washington State University (WSU) [attached]. Such research can be summarized in the following four points:

[Washington State University's] proposed adjustments to the USDA Rules require that USDA develop a program which incorporates the following four critical elements:

5. *A sampling program based on using a homogenized sample that consists of 25% -30% flower and the remainder being made up of stem/leaf and stalk.*
6. *A testing program that is at least 30 days before harvest or a post-harvest test utilizing an HPLC platform for compliance.*
7. *The sampling and testing Measurement of Uncertainty (MU) has variables requiring a 30%+ allowance.*
8. *Drying is part of harvesting, therefore we need an allowance to move a crop from field to drying facility prior to testing results even if that entails moving the crop between independent license holders.*

Alongside us, sharing a primary preference for post harvest compliance testing and a secondary preference for 30-day post harvest method, include the Oregon Department of Agriculture, the Washington State Department of Agriculture, Oregon State University and Washington State University.

2: Sampling Methodology—Flower vs. Whole Plant

AWHP stands by the extensive comments pertaining to the necessity for whole plant testing to accurately assess hemp for compliance. Since we submitted our previous comments, Washington State University has supplied evidence in favor of this method [attached].

3: Liquid Chromatography Factor, 0.877

AWHP supports High Performance Liquid Chromatography (HPLC) as the current gold standard for cannabinoid analysis of hemp. Other methods yet lack the veracity of HPLC. Until the emergence of a more effective method for analysis and as long as there lacks a standard validation protocol, USDA ought to utilize HPLC. Washington state is currently conducting research in this realm.

4: Disposal and Remediation of Non-Compliant Plants

AWHP maintains that there simply is no need for destruction of "non-consumable," or "non-ingestible" material. If a field, or crop, is found to be non-compliant, AWHP supports alternatives to destruction. Such alternatives might include accepting processes for remediation of THC, opportunities to route a "hot" crop into a non-consumable or non-ingestible product, or other means to mitigate risk for the cultivator. So long as the line between agricultural commodity and illicit material exists on a razor's edge, a cultivator should have viable opportunities to avoid destruction.

5: Negligence

Negligence must be proved beyond a shadow of a doubt. If a farmer purchases material which was procured with a reasonable expectation of compliance, they should not be punished. Punishment should be reserved for gross negligence. The hemp industry remains in its infancy. Until hemp is comprehensively understood and production of a crop of hemp can reliably test beneath said limit and levels of tolerance, there should remain a reasonable standard of forgiveness as to not unduly punish entrepreneurially spirited cultivators.

6: Interstate Commerce

AWHP continues to work with local authorities in Oregon to negotiate solutions that work for the industry and for regulators alike. We need to establish a stout tracking program so that inspectors in the field can easily verify compliant hemp which does not cause undue burden for private enterprise or for federal and state regulators. Current tracking processes for state-based, adult-use cannabis programs are too onerous at this juncture. Nor are they appropriate for hemp, as hemp is a federally recognized agricultural commodity and adult-use cannabis is not.

It is our belief that post harvest compliance testing would best alleviate the current burdens because plant material would be tested and verified as it would exist in the marketplace. AWHP continues to collaborate with pertinent parties to find effective and efficient options and looks forward to negotiations to come. Until then, we advise reference to precedential standards that currently already exist in the United States, such as the hemp tracking system recently established in Indiana and the barcode system for the USDA seed program. These protocols provide simple proforma solutions for the type of tracking appropriate for hemp. Similarly, the bonding programs and commercial agricultural merchant licenses utilized in Montana and Washington state may also prove useful precedents. Moreover, farmers and manufacturers will already have bills of lading, compliant certificates of analyses, and agricultural licenses/registrations etc. as matter of course in the trade and such documentation can be reasonably utilized for tracking purposes.

7: Hemp Seedlings, Microgreens, and Clones

Refer to our sentiments on end-user testing.

8: Hemp Breeding and Research

The hemp industries' growth regarding breeding and genetics depends on the ability and resources of the Universities in conducting ongoing research in a manner conducive to budgetary constraints. We point especially to the work currently being done by Washington State University (WSU) Institute of Biological Chemistry and Oregon State University (OSU) Global Hemp Innovation Center regarding this matter. For such endeavors to be conducted smoothly and effectively there need to be clear and absolute requirements and protocols for the destruction (if required by USDA IFR) of non-compliant crops.

9: Sampling Methodology—Homogenous Composition, Frequency, and Volume

We agree with sampling for compliance analysis based on end-use; however, we maintain that a postharvest sampling process of homogenized, whole-plant material is most accurately indicative of the identity of the plant and its constituents in as close to their natural proportions. Without this ideal process, analysis based on end-use is appropriate, but such standards should be universal to facilitate interstate (and hopefully international) commerce.

Again, as is standard for nearly all agricultural commodities and in the spirit of mitigating undue burden for the cultivator, when deciding how many plants are sampled from each acre or each field, we insist that research is still needed to establish what is appropriate and that only tentative requirements are established until such proper research is conducted. Especially considering large-scale operations, which is the appropriate standard to consider as the industry matures, we must be diligent in prioritizing what is necessary and pragmatic. At

this point, at least ten plants per acre for plots up to ten acres is appropriate according to industry sentiment and regulator experience. Moreover, the amount should always remain as minute as necessary according to what is duly representative and realistic.

10: Sampling Agents

Naturally, everything is contingent on establishment of methods before a training protocol is established. Until then, we would prefer resources are allocated elsewhere.

11. DEA Laboratory Registration

DEA involvement is unnecessary, and we should follow established standards for THC destruction as found in current hemp programs such as those in Oregon, Colorado, and Washington.

II. USDA/AMS Standards Program for Hemp-Derived Extract Concentrate

As is well known, during the hemp extraction process leading to final hemp-derived products (including food, beverage, and dietary supplement products), it is not uncommon for the extracted oil to rise about the 0.3% THC limit set out in the 2018 Farm Bill before being diluted back to 0.3% THC in the final product. The DEA IFR made explicit that DEA believes it has jurisdiction over the extracted oil if it goes above 0.3% THC.

We will address our concerns regarding the DEA IFR in the appropriate channels, but for the purposes of this comment, we maintain that it is USDA and not DEA that must establish a program to set standards for hemp-derived products while in- process, just as USDA did in the case of orange juice concentrate. We request that AMS establish a standards program for hemp-derived products similar to that established for extract/concentrate products, including orange juice from concentrate. **It is USDA- not DEA-that has experience with setting standards for in-process standards for food and beverage products.**

In the case of orange juice from concentrate, USDA has set standards and certified production of a product that is extracted, concentrated, and then diluted. In the case of orange juice concentrate AMS established a Brix value:

- Brix means the total soluble solids as determined when tested with a Brix hydrometer and applying the applicable temperature correction. The Brix may be determined by any other method that gives equivalent results.
- Brix/acid ratio means the ratio of the degrees Brix of the juice to the grams of anhydrous citric acid per 100 grams of the juice.
- Brix value means the refractometric sucrose value determined in accordance with the International Scale of Refractive Indices of Sucrose Solutions and to which the applicable correction for acid is added. The Brix value is determined in accordance with the refractometric method outlined in the Official Methods of Analysis of the Association of Official Analytical Chemists.
- Brix value/acid ratio means the ratio of the Brix value of the concentrate, indegrees Brix, to the grams of anhydrous citric acid per 100 grams of concentrate.[i]

For this program, USDA also established a system for inspection, certification and lab testing.[ii]

Pursuant to the AMS Concentrated Orange Juice Program, USDA has experience establishing complex science-based standards.[iii]

In parting, AWHP would like posit ourselves in full support of the comments submitted by Washington State University (WSU) Institute of Biological Chemistry, Washington State Department of Agriculture (WSDA), Oregon Department of Agriculture (ODA), Oregon State University (OSU) Global Hemp Innovation Center and the National Farm Bureau.

Thank you for your attention to these matters and we look forward to continuing to work with you to establish a strong and viable hemp and hemp-derived products industry.

Sincerely,

Dylan Summers

President, Association of Western Hemp Professionals
Lazarus Naturals OR

On behalf of the Board of Directors:

Bonny Jo Peterson, Industrial Hemp Association of Washington, WA
Bill Cyr, Unique Food Works, OR
Megan Duvall, FSOil, OR
Kelly O'Conner, Columbia Food Laboratories, OR
Jeff Beadle, Valley Industrial Services, OR
Clarence Laub, Dakota Hemp Co., ND
Nathan Howard, East Fork Cultivars, OR
Katey Herland, Canyon County Hemp and Hemp & Hops, CO
Jacob Crabtree, Columbia Valley Hemp, Co.
Jamie Fitterer, Montana Hemp Co., MT

cc:

Bill Richmond, Branch Chief, U.S. Domestic Hemp Production Program, Specialty Crops Program, AMS, USDA

Patty Bennett, Director, Marketing Order and Agreement Division, Specialty Crops Program, AMS, USDA

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[i] https://www.ams.usda.gov/sites/default/files/media/Canned_Orange_Juice_Standard[1].pdf
[ii] https://www.ams.usda.gov/sites/default/files/media/CommoditySpecificationforFrozenFruitJuices.pdf
[iii] §52.1321Explanation of analyses.

(a)The measurement of Brix value is determined on the thawed concentrate in accordance with the refractometric method for sugars and sugar products, outlined in the Official Methods of Analysis of the Association of Official Agricultural Chemists.
(b)Acid calculated as anhydrous citric acid, is determined by titration with standard sodium hydroxide solution, using phenolphthalein as indicator.
(c)Recoverable oil is determined by the following method:

METHOD

(1) Reagents.

Standard bromide-bromate solution-prepared and standardized to 0.099Nin accordance with Chapter 42, Standard Solutions in the current edition of the AOAC. For use, add 1 volume of standard solution to 3 volumes of water1to make 0.0247N solution. 1 ml. of 0.0247N solution supplies bromine to react with 0.00085g., or 0.0010 ml. of d-limonene. The solutions are stable for 6 months

2-Propanol-Reagent grade ACS (American Chemical Society).

Dilute hydrochloric acid- prepared by adding 1 volume of concentrated acid to 2 volumes of water.
Methyl orange indicator-0.1 percent in water.
lazzynatZ123!

(2) Apparatus.

Electric heater-with recessed refractory top, 500-750 watts.

Still,all glass- 500 ml. distillation flask with 24/40 standard taper neck; 200mm. Graham condenser with 28/15 receiving socket and drip tip; connecting bulb and adapter as shown in Figure 1.

Burette-10ml. or 25 ml. graduated to 0.1 ml., with easily controllable flow to permit both rapid and dropwise titration.

(3) Determination

(i) Pipette25 ml. of well-mixed sample (juice or reconstituted juice) into the distillation flask containing carborundum chips or glass beads and add 25 ml. of 2-Propanol.

(ii) Distill into a 150 ml. beaker. Continue distilling until solvent ceases to reflux then remove the flask from the heater.

(iii) Add 10 ml. of dilute hydrochloric acid and 1 drop of indicator. (An alternative method would be to prepare a solution containing 5 ml. of indicator and 1,000 ml. of dilute hydrochloric acid-then add 10 ml. of this acid-indicator mix to the 150 ml. beaker).

(iv) Titrate with the dilute bromate solution while stirring. The major portion of the titrant may be added rapidly, but the endpoint must be approached at about 1 drop per second. Disappearance of color indicates the endpoint.

(v) Determine the reagent blank by titrating three separate mixtures of 25ml. 2-Propanol and 10 ml. of dilute hydrochloric acid with indicator-without refilling the burette. Divide the total ml. of titrant used by three to obtain the average blank. Subtract the average blank thus obtained from the ml. of titrant used to titrate the distillate.

(vi) Multiply the remainder by 0.004 to obtain the percent recoverable oil by volume in the juice sample.

(vii) The ml. of recoverable oil per 100 grams of the concentrate is determined as follows:[percent oil by volume recovered] 4ml. recoverable oil per 100 grams concentrate =specific gravity of the concentrate

https://www.ams.usda.gov/sites/default/files/media/Frozen_Grapefruit_and_Orange_Juice_Standard%5B1%5D.pdf