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Cannabis Science and Technology Announces Editorial Advisory Board

Cannabis Science and Technology is pleased to announce the launch of its editorial advisory board (EAB). The distinguished members of the EAB will play a crucial role in defining the editorial direction and maintaining a high level of editorial quality and integrity.

The official charter of the advisory board for Cannabis Science and Technology is to support the publication in identifying the most significant developments in the cannabis science field, and in ensuring the relevance and technical accuracy of our content. In doing this work, board members support our mission to educate the legal cannabis industry about the science and technology of analytical testing and quality control. We are pleased to announce the following board members:

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For more information about our esteemed board members, please see the full announcement on our website: www.cannabissciencetech.com/news/cannabis-science-and-technology-announces-editorial-advisory-board.

University of Connecticut to Offer Cannabis Horticulture Course

The University of Connecticut (UConn, Storrs, Connecticut) announced plans to offer a class on cannabis horticulture through the Department of Plant Science and Landscape Architecture (1).

The course will be called “Horticulture of Cannabis: From Seed to Harvest” and will be taught by Gerald “Gerry” Berkowitz, a professor of plant science, and experts working in the cannabis industry today such as CEOs from successful business operations, for example, licensed cannabis growing facilities in Connecticut and cannabis testing laboratories.

According to UConn Today, the course will have a firm focus in horticulture, serving as a gateway to the entire cannabis industry. The introductory course will be open to all UConn students, regardless of their major, and has no prerequisites for enrollment. In addition to Berkowitz, expert guest lecturers will present on topics such as cannabis genetics, seed selection, soil and tissue testing, plant hormones, and laboratory testing of harvests.

Berkowitz currently has the ability to work with low tetrahydrocannabinol (THC) hemp cannabis plants, which will be used as examples for class demonstrations. As reported by UConn Today, the plants will be grown in concert with the course to provide students with hands-on examples of the range of horticultural methods applied to cannabis, such as propagating clones through cuttings, transplanting, training plants to alter canopy and flower architecture, culling males, pest management, and more. The course will be offered in the Spring 2019 semester (2).

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1) https://today.uconn.edu/2018/10/cannabis-course-responds-industry-need/.
2) http://www.plantscience.uconn.edu/.

Cannabis Science Conference Announces East Coast Show

The organizers of the Cannabis Science Conference announced a new east coast show for 2019. Cannabis Science Conference East will take place April 8–10, 2019, at the Baltimore Convention Center in Baltimore, Maryland. The flagship conference in Portland, Oregon will take place September 4–6, 2019.

Oral and poster abstracts are now being accepted for Cannabis Science Conference East. Please visit www.cannabisscienceconference.com for more information on both events.
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Error, Accuracy, and Precision

In the science of analytical chemistry, we quantitate things such as weights and concentrations. All of these measurements generate a number, but to truly understand the quality of data we have to know the size of the error in the measurement. Well known measures of data quality include accuracy and precision. Do you know the difference between these two metrics? Do you know how to quantitate them? And how does all of this apply to cannabis analysis? Please read on to find out more.

Brian C. Smith

A ny time a quantity is measured, be it your weight on a bathroom scale, the speed of light, or potency of a cannabis bud, there will be error involved in the measurement. Sources of error include environmental changes, power fluctuations, electronics, and good old fashioned human error. Error exists because human beings are not gods and thus cannot control all the variables all the time for any given measurement (1). Two of the most important types of noise are random noise and systematic noise. Let’s discuss random noise first.

Random Noise

Random error is caused by variables we cannot control as mentioned above. The sign of random error is random, that is, it is equally probable to be positive or negative. This is why measurements are often times expressed as X±y, where X represents the value of the measurement and y represents the amount of error in the measurement. Error is sometimes called noise, and the quality of data can be expressed as a signal-to-noise ratio (SNR) defined in equation 1.

\[
\text{SNR} = \frac{\text{Signal}}{\text{Noise}} \quad [1]
\]

The SNR concept is perhaps best illustrated using a cell phone call. In this case, the volume of the caller’s voice is the signal and the static in the connection is the noise. If the volume of the caller’s voice is large compared to the static, the connection has a good SNR and you can clearly hear what the other person is saying. Alternatively, if the static in the connection is high, and the caller’s voice can barely be heard above it, the SNR of the call is low and you will have trouble understanding what your caller is saying. Note that a high SNR phone call, or any high SNR data, will carry a lot of information. Whereas a low SNR phone call or low SNR data will carry very little information. This is why SNR is a measure of data quality.

In analytical chemistry error is often seen as “fuzz” in the baseline of chromatographic and spectroscopic measurements. An example of this is seen in Figure 1.

Figure 1 shows noise measured by a Fourier transform-infrared (FT-IR) spectrometer. Since the sign of random noise is random, the baseline fluctuates up and down randomly. The size of these wiggles is a measure of the noise (2). Note in Figure 1 that the size of the noise varies with wavenumber, which is typical of any spectrum measured using light (electromagnetic radiation).

The big peak at 2350 cm⁻¹ in Figure 1 is an artifact, which is a peak or signal in your data that is not from the sample. This peak is from the presence of unwanted atmospheric carbon dioxide inside the instrument. If you see a CO₂ peak in an FT-IR spectrum that you measure, it is an artifact.

A measurement of a signal-to-noise ratio, of course, needs a signal. In chromatography, spectroscopy, and other analytical techniques the signal is the magnitude of the measurement made. This is illustrated in Figure 2.

The y-axis in Figure 2 is in absorbance units (AU), which is a measure of the amount of light absorbed by a sample. The size of the peak at 461 cm⁻¹ in this case is the signal, which has a magnitude of 0.0215 AU. A measure of the error in a signal is called the peak-to-peak noise (PPN). In Figure 2, this is calculated by taking a section of the baseline and taking the highest noise point, in this case 0.01388 AU, subtracting from it the lowest noise point, which is 0.001144 AU, and obtaining a PPN of 0.00244 AU. The signal-to-noise ratio is then (0.0215 AU)/(0.00244 AU) for a value of ~9. This is not a particularly good SNR. Many laboratories measure what they call a limit of detection (LOD), which is typically calculated as 3x the noise in a measurement or an SNR of 3. By calculating an LOD a laboratory is saying, “this is the minimum signal I can reliably measure.”

Many cannabis laboratory pesticide
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analysis reports, for example, contain an LOD that gives the minimum amount of a pesticide whose presence can be reliably confirmed.

However, signals at or just above the LOD are too noisy to give reliable quantitative results. So, many laboratories use a limit of quantitation (LOQ) of 10x the noise level or an SNR of 10. By calculating an LOQ a laboratory is saying, “this is the minimum amount of signal with which I can give a reliable quantitative measure.” The LOQ in a pesticide report from a cannabis analysis laboratory tells you the minimum amount of pesticide that can be reliably quantitated in a sample. Thus, the SNR of 9 in Figure 2 is above the limit of detection but below the limit of quantitation.

Random noise can be reduced by observing the same quantity multiple times and then averaging those readings. The SNR improves as the square root of the number of observations, $N$, averaged as such:

$$\text{SNR} \propto \sqrt{N}$$  \[2\]

where $N$ is the number of observations averaged.

In essence, equation 2 works because random noise cancels itself as $N$ increases. Equation 2 is why we prefer to measure an average instead of a single observation, the average has the better SNR. Random error cannot be eliminated, but it can be minimized by controlling error sources and averaging data.

**Systematic Error**

Systematic error occurs when a measurement is consistently wrong by the same amount and in the same direction. A classic example of systematic error is a clock that has been not been set ahead for daylight savings time. This clock will always be 1 h behind whenever you look at it. It is an example of systematic error because it is always off by the same amount, 1 h and in the same direction, always behind.

To detect systematic error, one must have a true or reference value with which to compare your observation. In the United States the reference time value is supplied by the atomic clock at the National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland (3). Unlike random error, systematic error can be eliminated, in this case by simply setting the clock forward 1 h.

**Accuracy and Precision**

Accuracy and precision are terms in common use, and they are commonly confused. Some people seem to think they are the same thing. They are not.

**Precision** is a measure of the scatter in a set of measurements.

**Accuracy** is a measure of how far away a set of measurements is from the true values. This is illustrated in Figure 3.

Imagine you are tasked with weighing a 1.0 g standard weight on the same scale seven times. Because of error you will not get the same reading each time, but a spread of values. This is illustrated to the left in Figure 3. The bullseye represents the known value, 1.0 g, and the seven dots represent the seven readings.
you obtained. These readings are a random scatter and they are not particularly close to the bullseye; this data set is imprecise because the readings are widely scattered and not reproducible. This data is inaccurate because of how far on average the readings are from the bullseye, the true value. If your seven readings are about the same value, let’s say 1.9 g, they would form the plot seen in the middle of Figure 3. The readings form a tight cluster in the upper right hand corner of the bullseye, but their center is far from the true value of 1.0 in the center. This data set is said to be precise but inaccurate. It is precise because the scatter in the data is small and the points cluster tightly together. Precision is a measure of the amount of random error in a dataset. It is found by measuring the same value on the same sample multiple times. Precise data will give a wide scatter, like to the left in Figure 3. Precise data will be tightly clustered as seen in the middle of Figure 3. However, this data is clustered around 1.9 g, far away from the true value of 1.0 at the center of the bullseye. Thus, this data set is inaccurate because the points fall far from the true value. Precision is a useful measure of data quality since it quantitates random error, but it ignores systematic error so it is not a complete picture. The systematic error in the middle of Figure 3 is the distance between the center of the data cluster and the bullseye.

Accuracy is a measure of the amount of random and systematic error in a data set. If the seven weights you measure all cluster tightly around 1.0 g, the diagram at the right in Figure 3 will be obtained. This data is precise because the spread of the readings is small, and it is accurate because the points cluster tightly around the true value. This is the ideal situation. Accuracy is a better measure of data quality than precision since it incorporates both random and systematic error.

Now, if one does not have a true or reference value to compare to, let’s say you lose your 1.0 g standard weight, repeated measurements on some other sample can be made to obtain a precision value. For example, you might want take a pencil and measure its weight seven times. The
spread of these values will give you the precision of the balance. This is not as good as an accuracy determination, but since we do not have a standard weight, and we do not know the true weight of the pencil, the precision is the best we can do. When reporting error size in the literature, always make sure to disclose whether you are reporting an accuracy or a precision, and for an accuracy result always clearly state the source of the true value used in the calculations.

Is True Accuracy Achievable in the Cannabis Industry Right Now?
As mentioned above, obtaining an accuracy calculation requires a sample with a known value to be measured. For many industries, where plant material is processed such as tea, there exist NIST traceable standard reference materials (SRMs). For example, there exist SRMs for tea and tea extracts that can be used to give reference values for accuracy calculations (4). Since cannabis is not legal at the federal level in the United States, NIST, being a Federal agency, cannot develop cannabis SRMs for any of the materials that cannabis laboratories test. This has left the industry in the difficult position of having to develop its own standards and analytical methods.

Now, for chromatographic methods there are pure cannabinoid standards that can be used for calibration. However, since there are no SRMs available for the actual types of samples analyzed, such as cannabis buds and extracts, the effect of sample preparation on the final results is not reflected in the accuracy calculations performed. Thus, I have to conclude, perhaps controversially, that in the absence of the proper SRMs, true accuracy measurements in the cannabis analysis industry may not be possible.

This issue is reflected in the problem of inter-laboratory variation. This has been documented before and continues to be an issue (5,6). In my view, part of the problem is the lack of industry standard methods for cannabis analysis. Because of the lack of this guidance, laboratories have had to fend for themselves to develop their own methods. Thus, different laboratories have developed different ways of doing things, and this leads to different results. In particular, I have observed that many laboratories prepare samples in completely different ways, leading to variations in the results obtained.

All of this is not to say that cannabis laboratories are not precise, they are. They can get impressively reproducible data on a given sample. But until SRMs and standard methods are available, the search for a true accuracy and a solution to the inter-laboratory variation issue will, I believe, be problematic. In the meantime, it might be best for cannabis businesses to find a laboratory they trust, submit their samples to them, and only compare numbers from this chosen laboratory to themselves going forward.

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About the Columnist
Brian C. Smith, PhD, is Founder, CEO, and Chief Technical Officer of Big Sur Scientific in Capitola, California. Dr. Smith has more than 40 years of experience as an industrial analytical chemist having worked for such companies as Xerox, IBM, Waters Associates, and Princeton Instruments. For 20 years he ran Spectros Associates, an analytical chemistry training and consulting firm where he taught thousands of people around the world how to improve their chemical analyses. Dr. Smith has written three books on infrared spectroscopy, and earned his PhD in physical chemistry from Dartmouth College.
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Curbing the Cannabis Industry’s Appetite for Energy

Cannabis cultivation has a problem, a big problem: the electric bill! Earlier in my cannabis career, I heard growers complain about the cost of electricity eating into their profits—but now the situation is much worse, while the stakes are far greater. Energy consumption is under greater scrutiny than ever by regulators because of concerns over fossil fuels hastening climate change and the cost of expanding the grid. This means the cannabis industry better find solutions to cut down on its power use, or else the government will—and no one wants that.

Roger Kern

Just how bad is the energy problem in the cannabis industry? Energy inputs are estimated by several studies to be perhaps as much as half of total indoor cannabis cultivation costs. Based on this estimate, let’s assume 2000 kWh per pound of medicine and a typical energy cost of $0.24 (1). For growing a single pound of cannabis, that’s $480 worth of energy! And just what is this energy used for? It’s not just lighting; also sucking up power are air conditioners and chillers, dehumidifiers, carbon and high-efficiency particulate air (HEPA) filters, fans, and system controllers. In a 2016 study done for investor-owned San Diego Gas & Electric, Emerge Economics broke down the energy consumption for indoor cannabis production and found that lighting accounts for 38% of the energy consumed, venting 30%, and air conditioning 21% (2). Furthermore, when one considers the current means of energy production, the cultivation of 1 kg of cannabis results in the production of 4600 kg of carbon dioxide.

Clearly, there is plenty of room for improvement and, just as fortunately, there are many ways to improve. For now, let’s just talk about two: the cultivation structure and how it is illuminated. According to BOTEC Analysis, indoor cultivation has the highest energy consumption levels with 4400 kWh, 6100 kWh/kg. Greenhouses have much lower energy demands with 6 kWh, 580 kWh/kg (3). (BOTEC’s study considers the energy consumption of outdoor cultivation to be minimal.) This is why I recommend to cultivators that they start their site and structure selection process by giving serious consideration to a greenhouse.

In California, my home state, the Department of Agriculture has projected the following trends: There will be a shift in production from the northern to the southern part of the state while the use of controlled-environment agriculture (CEA) is forecast to jump dramatically over 2–3 years from 16% to more than 50% (4). I expect there will always be indoor, greenhouse, and outdoor production, and there are advantages to all these methods. Still, I would like to see the percentage of greenhouse facilities increase for the reduction of energy use while still enabling a controlled environment for maximum yield and quality.

Light emitting diodes (LEDs) are finding their way into more and more grows, despite skepticism by cultivators. Most of the controversy centers around sufficient power during the flowering phase and the nonsolar nature of the spectrum. Both concerns are not issues in the greenhouse, where LEDs serve to augment the solar spectrum during off-peak times of solar illumination such as early morning, late afternoon, winter months, and cloudy days. However, the energy benefits are real and substantial: Most manufacturers claim that their 600-W fixture can replace a 1000-W high-intensity fixture, resulting in a 40% reduction in lighting costs. This efficiency is further compounded by energy savings associated with cooling because of the heat produced from lighting; therefore, these practices are being adopted by CEA vegetable growers on a global scale. The reason is simple: LEDs save money by saving energy in the CEA setting while increasing yield and quality.

As the acceptance of cannabis spreads across the nation and world, it’s logical to assume more will be grown, which, of course, means an increasing demand for electricity. The industry would be wise to demonstrate its responsibility by acting now to cut back on power consumption. Because if cultivators don’t, strangers may be more than willing to act for them—and when they do, they may not have the industry’s wellbeing in mind.

An Expert’s Point of View

When looking for solutions to the cannabis industry’s high energy consumption, it’s necessary to have experts who are familiar with both cultivation and how utilities can help growers. Dan Duran has that knowledge, having worked for power companies and, as a professor, having researched cultivators’ use of power. Dan spoke with Cannabis Science and Technology magazine to share how the two sides can work together.

What has been your background and experience with the cannabis sector?

Dan Duran: I am a veteran of 25 years in the corporate world, where I led operations and marketing teams in the IT and...
energy sectors, including stints with American Telephone and Telegraph, Nippon Electric Corp., Southern California Edison, and Sempra Energy. I decided, after the tragic events on September 11, to move into academia and have been a faculty member at a California liberal arts college since then. At Whittier College, I study domestic and international sustainable development with a focus on energy and renewable resource management. I founded Energized Solutions, LLC, in 2002 with the goal to provide real-world data and research pertaining to sustainable development and renewable resource management to be used in and out of the classroom. Dr. Cinzia Fissore, who joined Energized Solutions a few years ago, and I have planned and executed several field-based research projects pertaining to agriculture for the California Public Utilities Commission (CPUC) and California-based electric, natural gas, and water utilities. We have conducted more than 200 field visits with a broad spectrum of growers, from row crops to dairy, nuts, and vineyards. Our goal is to better understand current practices, help the utilities and other stakeholders learn more about their customers, and develop and deliver programs to improve energy–water efficiency and other sustainability-focused programs and technical services. Over the last few years, we have tightened our focus on the urban agriculture sector and more specifically on CEA and greenhouse operations, especially the growth in Southern California.

You mentioned your energy–water work with utilities and the agriculture sector. What are the utilities thinking and doing with respect to the legalized and fast-growing cannabis business in California, and specifically with the increasing focus on CEA and greenhouse production by growers?

Duran: The good news is that a few utilities have recognized the need and opportunity to work with CEA and greenhouse growers, including those in urban areas. The case is that the majority of the utilities have opted to take a “wait and see” position. As a veteran manager of several utilities, the reality is that utilities are far more risk-adverse than other firms in the private or public arena, and while most of them highlight their commitment to serving customer needs, their primary goal is to protect investors by heeding to the dictates and regulations of the CPUC or equivalent oversight agency. For example, while the major utilities in California have dedicated agriculture account executives or service representatives that work with the large growers and post-harvest processors that have significant energy and water needs, the unfortunate case is that they rarely, if ever, interact, and know very little about the needs of CEA and greenhouse growers. Another factor is that many of those growers have operated in a “gray” area to elude the detection by utilities and most often take the “fast” in–out operations way rather than a systems approach. I have spoken to several of the utilities and the large consulting firms that help them with strategic planning and program evaluation, and while there are a few field people who recognize the need to learn and support growers, the senior management teams have almost consistently chosen to take limited initiatives. This is not a good situation, as the growing number and size of CEA and greenhouse growers need to better understand and take advantage of “Time of Use rates,” energy-efficiency programs and incentives, service planning infrastructure support, technical training on current and innovative technologies, and a broad spectrum of other services and support provided to the traditional medium to big users. Utilities increasingly want to be perceived as being environmentally responsible with strong corporate social responsibility agendas, but in this case, they have largely missed the opportunity to acknowledge the emergence of this new base of customers that are producing a high-value crop with significant social and economic benefits.

What should current and hopeful growers expect from the utilities and what is the most effective approach to use in securing technical and financial support from them?

Duran: Utilities are typically organized by function, such as distribution, energy–water efficiency, service planning, customer education, regulatory, and public relations. My first piece of advice is to do a bit of internet leg work and determine where the agriculture support team is situated and to contact the local account executive or service representative. While most of the utility agriculture teams are located in the traditional rural locales, they are the most likely to respond to a request for information on services. Growers should also identify the local or regional public affairs or equivalent representative because they are the most attuned to listening to users and their professional association advocates. The public affairs folks are charged to be the face of the utility to the local community and the local power structure, and they are responsible for articulating problems and opportunities to their utility management organization. While most of the mid- to larger utilities run education and training programs on efficiency strategies and new product offerings, they have yet to develop and offer programs customized to the needs of the CEA or greenhouse cannabis grower. With the exception of the recent 2017 Grower Report prepared by the Cannabis Conservancy for the Colorado Energy Office (5), there is a lack of solid and current data on the energy spends and efficiency levels of different energy-intensive (for example, lighting, heating, ventilation, and air conditioning [HVAC], and CO₂ generation) and water-operating modes used for outdoor and indoor operations. Still, it is commonly stated and believed that the energy spend constitutes between 30–45% of CEA-greenhouse grower operations. There is a real lack of understanding regarding which specific energy-use areas can
be best addressed via more energy-efficient technologies and what their impact is on the bottom line.

What should utilities put on their agenda to prepare and support the CEA-greenhouse growers?

Duran: The cannabis growers market has several key stakeholders, and the local energy, water, and waste utilities all have important roles to play in helping this industry mature with respect to energy efficiency, water management, and waste practices. At the moment, I am in conversation with several Southern California utilities about how best to prepare and support the CEA and greenhouse growers. The most important step is for the utilities to prepare and execute a data-gathering plan that identifies and records the current energy, water, and waste practices—what we call a baseline study. Utilities need to figure out where the near-future CEA-greenhouse growth will be with respect to their distribution systems, including both the electric and water distribution grids. Electric utilities tend to be “grid-focused,” and it is critical that they understand where the electric load will grow and how to best support it. The corollary need is for utilities to identify and work with the growers to provide a spectrum of services and support, from infrastructure service planning to information on how demand charges are calculated, the available time of use and other rate options, and the incentives and rebates for current and innovative technologies, including LED and virtual management systems. I believe that utilities will be motivated to respond to the growers who can provide data on plant-cycle growing needs, energy consumption by equipment and system, and operations practices. My hope is that the sustainably and profit-focused CEA sector and greenhouse growers will work with the utilities, equipment and tech providers, and consultants to plan and implement a triple bottom line strategy that generates value and simultaneously benefits people, the planet, and profits. It is my impression that utilities are very motivated to work with other sectors to promote leadership in efficiency and design (LEED) buildings and facilities that are both energy efficient and operationally advanced. My “call” to growers is to work with their local utilities (gas, electric, water, and waste), equipment and technology providers, and experienced consultants to lay the foundation for LEED growing operations that produce quality products using environmentally-responsible growing operations that increase bottom-line profits and generate value for both consumers and the cities and counties in which they operate.

Conclusion

We are entering a new era of cooperation and coordination between the cannabis industry and the public utilities. Reducing energy consumption and adopting more sustainable practices should become a high priority for cultivators. This will show the public and the utility companies that cannabis cultivators have a strong commitment to the environment, while still making a profit. This approach will generate a positive relationship with the utilities, create goodwill in the public eye, and demonstrate that cannabis cultivation is on the cutting edge of sustainability. By being innovative in cultivation practices and becoming leaders in lowering energy usage, cannabis cultivators will show the world that they are excellent stewards of our natural resources and good partners with public utilities.

References

3) M. O’Hare, D. Sanchez, and P. Alstone, “Environmental Risks and Opportunities in Cannabis Cultivation” (BOTEC Analysis Corporation internal publication, June 2013).

ABOUT THE INTERVIEWEE

Dr. Dan Duran is an Associate Professor at Whittier College where he teaches sustainable development. He is also the founder and principal of Energized Solutions, LLC, a CPUC-certified minority-owned business (MBE). His career includes senior planning and operations work with energy utilities and water agencies. His ongoing research and consulting focus is in the energy-water nexus, especially with the California agriculture community. He has met with hundreds of agriculture producers to identify their sustainability and best practices, and he works with utilities to optimize the potential for CEA cannabis production.

ABOUT THE GUEST COLUMNIST

Dr. Roger Kern is a scientist and technologist who cares deeply about the cultivation and health of plants in the cannabis industry. With his PhD in microbiology from the University of California, Davis, Plant Growth Laboratory, he solves the most challenging problems in hydroponics, from studying the root microbiome to developing nutrients and lighting systems to ensure plant health and a disease-free lifecycle. He spent 22 years at NASA’s Jet Propulsion Laboratory as a scientist, technologist, and research leader before becoming the President of Agate Biosciences, a consulting firm for project management, systems engineering, and science in CEA for the past eight years. He leads developments to optimize sustainability, consistency, quality, and yield without compromising plant health. Direct correspondence to: rkern@agatebiosciences.com
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Setting the Standard: Considerations When Handling DEA-Exempt Cannabinoid Reference Standard Preparations Used for Potency Determination

With the growth of the cannabis market, significant investments have been made into the setup, operation, and maintenance of cannabinoid potency testing laboratories. Results generated in these laboratories are not only dependent upon the accuracy of the testing methods and sample preparation, but on the quality of the reference standards from which batches of cannabis samples are directly quantified. The quality, consistency, and reliability of reference standard solutions are not only a priority for manufacturers, but it is in the best interest of testing laboratories to protect and maintain the integrity of these formulations during use and storage. In this manuscript, reference standard qualifications are discussed and techniques for qualifying incoming reference standards are suggested. Additionally, in-laboratory handling of reference solutions is explored to promote and preserve accuracy of the formulations during use.

Catharine E. Layton and Andrew J. Aubin

Currently in the United States, cannabinoid isolates are classified as Schedule I controlled substances under the Controlled Substance Act (CSA) Title 21 Code of Federal Regulations (CFR) Parts 1300 to 1308. The distribution of these isolates is controlled and monitored by the Drug Enforcement Administration (DEA) division of the U.S. Department of Justice (1). Purchasing and use of controlled substances must adhere to the requirements of the Controlled Substance Act and any state enforcement agency, as well as the terms and conditions of any Institutional Research or Analytical Licenses or Registrations issued in accordance with proposed laboratory activities. Sanctioned DEA-exemptions are granted for cannabinoid preparations after approval by the DEA and Local State Codes to registered manufacturers that supply these solutions for laboratory or scientific research purposes (2).

A reference standard is broadly described by the U.S. Food and Drug Administration (FDA) and U.S. Pharmacopeia (USP) as a well characterized, highly purified compound that serves as a specimen or performance calibrator from which drug substances, excipients, impurities, degradation products, or compendial reagents are identified and quantified for purity and potency. Any material can be called a “reference standard,” but the degree to which that material is characterized defines if the reference standard is an acceptable calibrator for the intended application. Simply because DEA sanctions have been provided to a particular reference standard manufacturer, it does not automatically ensure that the reference standards produced by that manufacturer meet the quality and reliability specifications for laboratories testing cannabinoid samples for potency.

Certified cannabis testing laboratories operate under International Organization for Standardization (ISO) and International Electrotechnical Commission (IEC) 17025 accreditations issued by the American Association for Laboratory Accreditation (A2LA) to provide confidence that results generated in these testing laboratories are high quality and consistent. Reference standards used by these laboratories are held to the same ISO quality standard, in addition to ISO 17034:2016, which focuses specifically on an evaluation of process uncertainty. This value is associated with manufacturing as a whole, and is reported along with orthogonal testing results, in the reference standard traceable documentation (certificate of analysis [CoA]) provided as per ISO 9001:2015. These ISO accreditations offer a foundation of integrity when selecting a manufactured cannabinoid reference standard material supplier.
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Evaluation of Incoming Reference Standards

Accurate determination of chromatographic purity depends upon the ability of an analytical method to resolve the analyte from closely eluted impurities. Since cannabis testing methods have yet to be standardized within the industry, the ability of chromatographic methods to separate the reference standard peak of interest from the impurities may not be consistent between laboratories. Because of this lack of standardization, chromatographic assay results determined for the reference standard solution in a sample testing laboratory may be different than the chromatographic assay value (mg/mL) reported in the CoA.

To demonstrate, three certified DEA-exempt ampouled cannabidiol (CBD) and Δ⁹-THC reference standards formulated in methanol (1.0 mg/mL) were purchased from different certified manufacturers (sources withheld). The chromatographic profiles of the CBD and Δ⁹-THC DEA-nonexempt isolate starting materials were compared to DEA-exempt ampouled reference standards using the same chromatographic platform (Figure 1) (3).

The CBD, Δ⁹-THC isolate starting materials, and the ampouled reference standards were confirmed to be spectrally pure by ultraviolet-photodiode array (UV-PDA) detection, and the mass was confirmed by mass spectrometry (MS) using the in-house validated chromatographic method. From the linear response of a CBD isolate reference standard, the concentration of CBD in the DEA-exempt ampouled preparations was calculated and compared to the CoA. Concentrations of CBD were 1.013 mg/mL, 1.001 mg/mL, and 1.001 mg/mL. All values were within the concentration range specified by the manufacturer CoA at 1.002 ± 0.011 mg/mL, 1.002 ± 0.006 mg/mL, and 1.000 ± 0.005 mg/mL, respectively. From the concentration values, it was concluded that although the references standard solutions contained different impurities, the in-house validated method provided a comparable chromatographic separation of CBD from the impurities when compared to the chromatographic assay method used to prepare the CoA.

Only two out of the three Δ⁹-THC DEA-exempt ampouled formulations were within the concentration range reported on the respective CoA when compared to the linear regression of the Δ⁹-THC isolate. The UV-PDA spectral purity and MS response for Δ⁹-THC in the formulation and the isolate were comparable. The reference standard formulation that lies outside the CoA concentration range does not immediately imply that the manufacturer provided an inaccurate reference standard concentration assessment. It is a reminder that qualification of an incoming reference standard should not be made based upon a single testing result. In this study, results were generated for only one reference standard vial. It is recommended that replicate vials are analyzed from the same lot, and that conclusions are made based on an average concentration determination and standard deviation value for the incoming lot.

Alternatively, an evaluation of acceptability can also be made by comparing the average response factor (RF) (equation 1) of the incoming reference standard to the RF observed in the historical data for previous reference standards of the same analyte. This approach requires that cannabis testing laboratories compile average RF and concentration information over time for incoming lots, and define acceptable incoming RF tolerance limits. With this technique, it is important to use the same chromatographic separation method, confirmed to be consistent and robust over time by fulfilling method system suitability criteria (that is, recovery and resolution).

Equation 1 shows the average response factor (RF):

\[
RF = \frac{\text{Peak area (μV * s)}}{\text{Concentration (mg/mL)}}
\]

![Figure 1: Overlay of DEA-nonexempt CBD and Δ⁹-THC isolate starting materials (black) with DEA-exempt ampouled reference standards from three manufacturers (green, red, blue) when separated using a validated chromatographic testing method for assay.](image-url)
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Preparation of Reference Standards for Sample Testing

In the testing laboratory, reference standards and sample solutions often require dilution of the initial stock solution to prepare a working standard. When working with solvent-based solutions such as cannabinoid formulations, it is important to select a pipette that will maintain accuracy. In this study, cannabinoid DEA-exempt and isolate reference standards were prepared via serial dilution using volumetric pipetting devices common in testing laboratories. Calibration curves for CBD and Δ²-THC were prepared using a glass volumetric pipette, an air interface pipette, and a positive-displacement syringe pipette using a 1.0-mg/mL cannabinoid reference standard formulation. Practical precautions were used to reduce error, such as using calibrated pipettes, prewetting the pipette tip with sample solution, working at a constant temperature, pipetting in a fully upright position, and avoiding transfer of the solution on the outside of the pipette tip to the collection vessel. The standard curves for linear regression were prepared to show the accuracy of each pipetting method.

Figure 2a exhibits the standard curve generated for DEA-exempt CBD reference standard using the air interface pipette. Although several of the data points in the serial dilution sequence appear to follow a linear trend, the curve was not linear by statistical regression analysis ($R^2 = 0.8763$). There is no justification to exclude the data points that do not statistically conform to the linear regression line ($y = mx + b$). The results were replicated by two analysts with two different air-displacement pipettes to confirm the nonlinear trend was consistent for this type of pipette.

In contrast, for calibration curves prepared with a positive-displacement syringe pipette, or a glass volumetric pipette, the data points were repeatedly statistically linear ($R^2 \geq 0.999$), as demonstrated for CBD in Figure 2b.

Differences in linearity between the pipetting methods can be understood by considering the mechanism of operation and how it is impacted by the physical properties (that is, density and vapor pressure) of the solution. For example, air-displacement pipettes rely upon the movement of a piston and calibrated air-cushion to regulate the volume measurement. Upward movement of the piston produces partial vacuum in the tip, which causes a metered amount of the sample to be aspirated into the tip. The air-cushion acts like an elastic spring from which the volume of liquid in the tip is suspended (Figure 3a). The volume of the air-cushion, which varies depending on the density, vapor pressure, viscosity, and temperature of the liquid being pipetted (4,5), is most often calibrated with distilled water as the test medium. Solvents with vapor pressure different from that of water, such as those utilized for commonly used cannabinoid solvents, push against the calibrated air-cushion forcing it to expand. The aspirated solution is then displaced from the pipette tip via dripping. This phenomenon compromises the accuracy and precision of the dispensed volume (5). Additionally, commonly used solvents for cannabinoids such as methanol, ethanol, or hexane have a lower density than that of water. Thus, the volume of liquid pipetted with an air-displacement pipette is systematically high when using these solvents neat, or as a part of a mixture, if the air-cushion pipette was calibrated with water.

The physical influences that impact an air-cushion pipette are not applicable to positive-displacement or glass pipettes. These pipettes repeatedly generated serial dilutions that were statistically linear. Positive-displacement pipettes operate using an integrated piston, or internal syringe. The pipette tip is immersed in the liquid and the piston rod moves down to displace the volume of air corresponding to the desired volume of the pipetted liquid. As the piston is released, the liquid is drawn into the tip. The piston comes into direct contact with the liquid, leaving no air-space (Figure 3b). This pipetting method is suitable for both

Figure 2: DEA-exempt CBD reference material standard curves generated using (a) air-displacement and (b) glass volumetric or positive-displacement pipettes. Replicate curves were prepared by multiple analysts to confirm linear regression trends.
solvent-based cannabinoid reference standards and highly viscous liquids, such as extracted cannabis oils.

When using glass volumetric pipettes, although the pipetted solution comes in contact with the air-space, the meniscus is visually titrated against a volumetric calibration mark. Delivery time and waiting time determine pipetting accuracy. The delivery time is the period of time after the suction is released for the meniscus to fall from the upper volumetric mark to the pipette tip. Waiting time begins after the liquid reaches the tip and comes to rest (6). Both must be adequately used to ensure accurate pipetting, no matter the physical properties of the liquid.

Storage and In-Use Considerations
Instrumentation and consumables are the largest expense when operating a testing laboratory. Consumables are defined as items that are used for an intended purpose and must be replaced because they wear out or are used up (7). They include both reference standards and sample testing vials (8). Cannabinoid reference standards are packaged in glass ampoules sealed under inert gas to promote stability by preventing evaporation and degradation from air and light during transport (9). After the ampoule has been opened in the laboratory, a key concern is the potential for degradation during general use. A decrease in purity because of chemical degradation is always a concern, but for purposes of this manuscript, the solutions were stored before use at the temperature recommended in the package insert.

It is common practice to transfer reference standard solutions from the shipping ampoules to liquid chromatography (LC) vials for use and storage. LC vials are typically manufactured from polypropylene plastic or glass. Although polypropylene vials may be more economical, surface-treated inert glass vials can be certified as chemically clean by the manufacturer. This certification minimizes the potential for chromatographic anomalies caused by manufacturing process contamination, and is especially important for samples intended for low-level impurity analysis by mass spectrometry.

The most common closures for these LC vials are caps that snap or screw onto the vial. Snap caps provide an advantage because they are easy and fast to assemble. Glass LC vials may show imperfections because of the manufacturing process. For example, cracks in the cap may result after stretching over an opening that is too large, or for a smaller opening, the cap may not provide an adequate air seal. Both result in the escape of the solvent from the reference standard solution, resulting in an increase in the cannabinoid concentration. Although it may take a few extra seconds to secure a cap with screw threads, the threads provide adequate mechanical force to hold the septum in place and provide a consistent air seal.

There is an overwhelming variety of cap septum offerings including
rubber, polyethylene, polypropylene, silicone, and polytetrafluoroethylene (PTFE). The challenge is determining which is appropriate for solvent-based cannabinoid solutions. Rubber and silicone alone are not recommended because they may express extractables and leachables when in contact with solvents. Chemically resistant polyethylene and polypropylene septa are intended for single injection, aqueous-based sample mixtures. Although polypropylene septa offer improved solvent compatibility compared to polyethylene, the piercing force is slightly higher, resulting in an increase in the probability of coring. Pure PTFE septa are not resealable and should not be used with highly volatile solvents, short cycle times, or multiple injection methods, all of which are common when testing cannabinoids. Laminated hybrid PTFE–silicone septa provide the ideal solution. A thin film of PTFE laminated to the side of the septum that faces the sample limits exposure of the silicone elastomer to the cannabinoid solvent, while the silicone on the outside surface provides resealing capability (10). Additionally, these hybrid septa are offered in a preslit format to eliminate vacuum formation or coring as it is pierced by the injection needle.

To demonstrate how quickly the reference standard solution can be affected by an inadequately sealed vial, a DEA-exempt CBD reference standard was removed from the sealed ampoule and aliquoted into two, certified, Acquity 12 x 32 mm sample manager LC vials (Waters Corporation). Both vials were sealed with screw-cap closures. One screw cap contained an inexpensive solvent-incompatible septum susceptible to coring (source withheld), and the other contained a preslit PTFE–silicone self-sealing septum (Waters). Replicate injections were performed from each vial over the course of 1.5 days while the vials were stored in the sample manager at 20 °C.

The DEA-exempt reference standard CBD concentration inside the vial that used the self-sealing PTFE–silicone septum remained stable after 1.5 days of replicate injections (Figure 4). A hole was observed in the less expensive, solvent-incompatible septum after collecting data at two injection time points. The coring resulted in evaporation of the reference standard solvent from the cannabinoid over 1.5 days, resulting in concentration of the solution. It is anticipated that a sample manager without temperature control capabilities would likely result in an even greater increase in solvent evaporation through the open septum.

It is not suggested that reference standard solutions are stored for long periods in LC vials with septum. They can be stored for longer periods in an LC vial with a chemically compatible septumless storage cap to reduce solvent loss (11). Even with this precaution, one must consider the number of times the vial is opened and closed to minimize solvent evaporation. As with incoming reference standards, the RF value can be monitored to determine if the standard is fit for use after storage based on its concentration.

Conclusions
Cannabis testing laboratories rely directly on solvent based DEA-exempt cannabinoid reference standards to accurately quantify cannabis samples. After selecting a reputable supplier, laboratories should consider adopting a qualification process for the incoming reference standards to ensure incoming formulations meet in-house assay specifications. Because cannabinoid reference standard solutions are solvent based, laboratories must be diligent when handling these formulations (that is, pipetting, vial storage) to promote integrity of the concentration from which potency determinations are established.

References

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Leveraging Selectivity and Efficiency to Take the Strain Out of LC–UV Method Development for Cannabinoid Profiling

More than 100 cannabinoids have been isolated from cannabis in addition to the five most commonly tested: Δ⁹-tetrahydrocannabinol (Δ⁹-THC), Δ⁹-tetrahydrocannabinolic acid (THCA), cannabidiol (CBD), cannabidiolic acid (CBDA), and cannabinol (CBN). Although many methods have been published that show the separation of these major cannabinoids, most do not take into account the possibility of interference from minor cannabinoids. This interference is most problematic in concentrates where minor cannabinoids can be enriched to detectable levels. Additionally, some terpenes absorb ultraviolet (UV) light at the same wavelength as cannabinoids, which can result in an additional source of interference. In this study, the liquid chromatography (LC)–UV separation of 16 cannabinoids of interest was performed while the potential impact from minor cannabinoids and terpenes on reported potency values was monitored. The method was applied to commercially available CBD oils that have recently become suspect because of inaccurate label claims.
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negative impact to the health and safety of consumers.

Although the state of analytical testing in the cannabis industry has improved since the inception of legalized medical cannabis, there are still many opportunities to update existing methods in this dynamic market. Historically, interest in cannabinoids typically focused on THC, CBD, and their carboxylated forms, but the analysis of more-abundant minor cannabinoids is starting to gather momentum. Testing laboratories most frequently use liquid chromatography (LC)-based techniques paired with ultraviolet (UV) detection for cannabinoids because of their hydrophobic and shape-selective characteristics. The combination of the appropriate stationary phase with SPPs increases the speed and improves resolution compared to traditional fully porous particles (SPPs) of the same particle size. The use of a 150 mm x 4.6 mm analytical column allows for the most efficient separations under the constraints of legacy instrumentation. Using isocratic conditions it was found that 14 of the 16 cannabinoids could be baseline resolved; however, CBNA was coeluted with Δ2-THC regardless of flow rate, column temperature, or mobile-phase composition. In an attempt to separate CBNA from Δ2-THC, the pH of the mobile phases was adjusted by using 0.1% acetic modified mobile phases in place of formic acid. This strategy was attempted in an effort to take advantage of the pKₐ of CBNA because it would be less retained in its ionized form, but THC would not be impacted as a neutral compound. Ionized compounds are more polar and not as strongly retained by reversed-phase mechanisms. The use of acetic acid maintained the same selectivity and resolution of the original separation, but still resulted in the coelution of CBNA and Δ2-THC. The pH of the mobile-phase system was further increased by the use of water modified with 5 mM ammonium formate and unmodified acetonitrile. This approach

Development of LC Method Conditions

Method development began with the goal of achieving baseline resolution for the following 16 cannabinoids by LC–UV: Δ9-THC, Δ⁸-tetrahydrocannabinolic acid (THCA), CBD, cannabidiolic acid (CBDA), cannabinol (CBN), cannabidivarinic acid (CBDA), cannabidivarin (CBDV), cannabigerolic acid (CBGA), cannabigerol (CBG), tetrahydrocannabinolic acid (THCVA), cannabinolic acid (CBNA), Δ⁴-tetrahydrocannabinol (Δ⁴-THC), cannabicyclol (CBL), cannabichromene (CBC), and cannabichromenic acid (CBCA). A 150 mm x 4.6 mm C18-type column packed with 2.7-μm superficially porous particles (SPPs) was initially scouted using popular mobile phases for the analysis of cannabinoids (water and acetonitrile modified with 0.1% formic acid) with UV detection at 228 nm. C18-type columns are ideal for the separation of cannabinoids because of their hydrophobic and shape-selective characteristics. The combination of the appropriate stationary phase with SPPs increases the speed and improves resolution compared to traditional fully porous particles (SPPs) of the same particle size. The use of a 150 mm x 4.6 mm analytical column allows for the most efficient separations under the constraints of legacy instrumentation. Using isocratic conditions it was found that 14 of the 16 cannabinoids could be baseline resolved; however, CBNA was coeluted with Δ²-THC regardless of flow rate, column temperature, or mobile-phase composition. In an attempt to separate CBNA from Δ²-THC, the pH of the mobile phases was adjusted by using 0.1% acetic modified mobile phases in place of formic acid. This strategy was attempted in an effort to take advantage of the pKₐ of CBNA because it would be less retained in its ionized form, but THC would not be impacted as a neutral compound. Ionized compounds are more polar and not as strongly retained by reversed-phase mechanisms. The use of acetic acid maintained the same selectivity and resolution of the original separation, but still resulted in the coelution of CBNA and Δ²-THC. The pH of the mobile-phase system was further increased by the use of water modified with 5 mM ammonium formate and unmodified acetonitrile. This approach

![Figure 1: The separation of 16 cannabinoids by HPLC–UV. Column: 150 mm x 4.6 mm, 2.7-μm Raptor ARC-18, mobile-phase A: water, 0.1% formic acid (v/v), 5 mM ammonium formate; mobile-phase B: acetonitrile, 0.1% formic acid (v/v), elution: isocratic at 75% B over 9 min; flow rate = 1.5 mL/min, injection volume: 5 μL; oven temperature: 30 °C; detection: UV absorbance at 228 nm. Peaks: 1 = CBDVA, 2 = CBDV, 3 = CBDA, 4 = CBGA, 5 = CBG, 6 = CBD, 7 = THCV, 8 = THCVA, 9 = CBN, 10 = CBNA, 11 = Δ²-THC, 12 = Δ⁸-THC, 13 = CBL, 14 = CBC, 15 = Δ⁹-THCA, 16 = CBGA.](image)
resulted in a loss of retention for all acidic cannabinoids because of the ionization of carboxylic acid functional groups. To reach an intermediate pH, the aqueous mobile phase was modified with both 0.1% formic acid and 5 mM ammonium formate, whereas the organic mobile phase was modified with only 0.1% formic acid. These mobile phases combined with a 1.5 mL/min flow rate using 75% organic allowed for the partial resolution of CBNA from Δ²-THC while still maintaining the separation of the remaining 14 cannabinoids. To this point, a column oven temperature of 40 °C had been used for method development. To enhance the shape selective characteristics of the stationary phase, the column oven temperature was reduced to 30 °C. This adjustment resulted in the baseline separation of all 16 cannabinoids with a complete cycle time of 9 min (Figure 1).

### Additional Sources of Interference

During LC method development, it was determined that minor cannabinoids can affect the quantitation of the major cannabinoids CBD and Δ⁹-THC. Another class of compounds found in cannabis, terpenes, can also be present in high enough concentrations to interfere with sample analysis. A mix of 21 common terpenes found in cannabis was prepared and

### Table I: Inter-run accuracy and precision (n = 6)

<table>
<thead>
<tr>
<th>Analyte</th>
<th>QC LLOQ 5.00 μg/mL</th>
<th>QC Low 30.0 μg/mL</th>
<th>QC Mid 150 μg/mL</th>
<th>QC High 400 μg/mL</th>
<th>QC Dilution (20-fold) 1000 μg/mL</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Avg. Conc. (μg/mL)</td>
<td>Avg. Accuracy (%)</td>
<td>% RSD</td>
<td>Avg. Conc. (μg/mL)</td>
<td>Avg. Accuracy (%)</td>
</tr>
<tr>
<td>CBD</td>
<td>4.96</td>
<td>99.2</td>
<td>3.14</td>
<td>30.8</td>
<td>103</td>
</tr>
</tbody>
</table>

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injected using the same conditions as previously developed for cannabinoids. Fortunately, the absorbance profile for most terpenes differs from cannabinoids such that they do not result in over reporting for cannabinoids. The terpenes that absorb UV light best at 228 nm, ocimene and its isomers, do not interfere with the quantitation of any monitored cannabinoid. Minor terpene interferences were found that could impact the quantitation of CBGA and THC-VA if present in substantial concentrations, but the quantitative analysis of these cannabinoids is not currently required in states such as California and Colorado (5,6).

**Sample Preparation**

The utility of the chromatographic method was evaluated by performing quantitative analysis of CBD in commercially available hemp-derived CBD oils. Calibration standards and quality control (QC) samples were prepared in 25:75 water–methanol across a linear range of 5–500 μg/mL. A QC dilution sample was prepared at 1000 μg/mL and diluted 20-fold to demonstrate the validity of diluting samples into the linear range. Six hemp-derived CBD oils were obtained for analysis. These products consisted of three vape oils, two sublingual oils, and one raw hemp oil. Next, 50 μL of CBD product was extracted using 950 μL of methanol followed by vortexing for 30 s at 3000 rpm. After that, 750 μL of the sample was then mixed with 250 μL of water followed by vortexing for 10 s at 3000 rpm. Finally, 400 μL of the sample was filtered using a Thomson Single Step standard filter vial with a 0.2-μm polyvinylidene fluoride (PVDF) membrane before LC–UV analysis.

**Results and Discussion**

Using linear 1/x weighted regression, the method showed good linearity for CBD with an r² value of 0.999. The method accuracy was demonstrated to be within 4% of the nominal concentration for all QC levels. The percent relative standard deviation (%RSD) was within 3.14% for all QC levels indicating good method precision (Table I). According to California regulations, a cannabis product must not differ from the labeled concentration of CBD by ±10% (5). Applying this labeling guideline to the samples evaluated in this study results in the possibility of all six products not being in compliance with these regulations (Table II). One product failure is attributed strictly to labeling nomenclature as the CBD content is not directly stated. Vape oils did not contain a significant amount of CBDA to account for the discrepancy in the labeling. The large discrepancy in the result for Product 3 is most likely because of photodegradation from improper packaging. In addition to CBD, other cannabinoids were found

---

**Table II: Determined concentrations of CBD in commercially available hemp-derived CBD oils**

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Product Type</th>
<th>CBD, Label Claim (mg/mL)</th>
<th>±10% Range (mg/mL)</th>
<th>CBD, Actual Concentration (mg/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Product 1</td>
<td>Vape oil</td>
<td>3.33</td>
<td>3.00–3.66</td>
<td>2.05</td>
</tr>
<tr>
<td>Product 2</td>
<td>Vape oil</td>
<td>6.67</td>
<td>6.00–7.34</td>
<td>5.59</td>
</tr>
<tr>
<td>Product 3</td>
<td>Vape oil</td>
<td>500</td>
<td>450–550</td>
<td>0.242</td>
</tr>
<tr>
<td>Product 4</td>
<td>Sublingual oil</td>
<td>11.0 (hemp extract)</td>
<td>NA</td>
<td>6.24</td>
</tr>
<tr>
<td>Product 5</td>
<td>Sublingual oil</td>
<td>6.11</td>
<td>5.50–6.72</td>
<td>5.21</td>
</tr>
<tr>
<td>Product 6</td>
<td>Raw hemp oil</td>
<td>30.0</td>
<td>27.0–33.0</td>
<td>18.4</td>
</tr>
</tbody>
</table>

---

**Figure 2:** The separation of 16 cannabinoids by UHPLC–UV. Column: 100 mm x 3.0 mm, 1.8-μm Raptor ARC-18; mobile-phase A: water, 0.1% formic acid (v/v), 5 mM ammonium formate; mobile-phase B: acetonitrile, 0.1% formic acid (v/v); elution: isocratic at 75% B over 4 min; flow rate = 1.0 mL/min; injection volume: 1 μL; oven temperature: 30 °C; detection: UV absorbance at 228 nm. Peaks: 1 = CBDA, 2 = CBDV, 3 = CBD, 4 = CBGA, 5 = CBG, 6 = CBD, 7 = THCV, 8 = THCVA, 9 = CBN, 10 = CBNA, 11 = Δ⁹-THC, 12 = Δ⁹-THC, 13 = CBL, 14 = CBC, 15 = THC, 16 = CBCA.
in most samples. Additional cannabinoids that were found included: CBN, CBDV, CBDA, CBG, Δ²-THC, Δ⁸-THC, CBN, CBNA, and CBC. The concentrations of these cannabinoids were not quantitatively evaluated because of their relative low abundance, but the concentration of Δ²-THC in all samples appeared to be well below 0.3% w/w based upon the peak height of a 50-μg/mL standard allowing the use of THC-free labels.

To ensure that sample results were not skewed because of poor extraction efficiencies, a recovery experiment was performed by spiking each product type with an additional 2.00 mg/mL of CBD. Recovery results for vapo oil, sublingual oil, and raw hemp oil were 102%, 98.5%, and 105%, respectively.

With regard to sample preparation, it should be emphasized that the addition of water following extraction with methanol is critical to precipitate lipids that are present in raw hemp oils and common carrier oils for sublingual products (that is, coconut medium-chain triglycerides [MCT] oil, sunflower oil, sesame oil, and so forth). Failure to perform this step will cause sample precipitation to occur once introduced into the mobile phase during high performance liquid chromatography (HPLC) analysis. The inlet frit of the analytical column will quickly clog resulting in elevated back pressures and decreased column lifetime. It is important to not combine the addition of water into the extraction step with methanol because this has been shown to result in reduced recoveries for cannabinoids in cannabis oils (7).

Ultrahigh-Pressure Liquid Chromatography Analysis

To improve laboratory throughput, the efficiency of sub-2-μm SPPs can be used to increase the speed of analysis while maintaining the same resolution. By pairing an appropriate system to a 100 mm x 3.0 mm analytical column packed with 1.8-μm SPPs of the same stationary phase, the separation can be achieved in 4 min (Figure 2). This approach results in a cycle time that is more than twice as fast with a 70% reduction in consumed mobile phase per sample. Columns must be effectively paired to an appropriate LC system that is not only robust at high system back pressures, but also optimized to reduce extra column volumes. Any dispersion that occurs in the valves, connecting tubing, and flow cell will reduce the efficiency and the resolution of separations. It is particularly important to use low-volume flow cells (approximately 1 μL) because of the relatively large volumes present in standard flow cells (8).

Conclusions

Considering the structural similarities of cannabinoids, it can be difficult to develop a robust analytical method that is capable of producing accurate results. Increasing interest in the analysis of minor cannabinoids can further complicate the separation. Fortunately, demanding separations can still be achieved through deliberate method development by leveraging selectivity and efficiency. The power of selectivity is realized through changes to the mobile phase that can alter the pH for a critical separation to occur or a change in oven temperature that can change the characteristics of a stationary phase. The increased efficiencies of SPPs compared to fully porous particles of the same particle size enables faster separations. Pairing sub-2-μm SPPs with appropriate instrumentation is critical for those laboratories interested in pushing the limits of sample throughput. It was demonstrated that 16 cannabinoids of interest can be separated in 9 min on a traditional HPLC system. The developed method was compatible for the identification of cannabinoids and quantitation of CBD in hemp-derived CBD oils. As interest in expanded cannabinoid profiles continues to grow, selectivity and efficiency can continue to be leveraged to remove the strain of LC–UV method development.

References

Certified Reference Material Manufacturing Challenges

Some analysts will look at a list of pesticides or solvents and visualize adding small amounts of pure material of each analyte into a volumetric flask and then bringing the flask up to volume with their solvent of choice. In rare instances this technique will work, but not often. Prior to making a mixture, factors such as solubility and reactivity must be considered. Are all of the analytes soluble in the solvent that I want to use? Do I need to use an intermediate solvent? Will the analytes react with each other or will they react with the solvents? Do I need to add a preservative? Which preservative should I use and will any of the analytes react negatively to the preservative? The answers to these questions come from experience and a working knowledge of the chemistry of reference material (RM) production. Manufacturing the standard mixture is only the first step in creating a certified reference material (CRM). The CRM must be characterized using a metrologically valid procedure. A certificate of analysis is produced listing certified values for specified properties and all analytes, including a calculation of total uncertainty, homogeneity, stability under specified conditions, and metrological traceability. The certificate of analysis is delivered with the standard.

Don Shelly

CRM Challenges
All CRM manufacturers strive for the lowest possible combined uncertainty during the manufacturing process. There are some variables that cannot be precisely controlled during the manufacturing process such as the limitations of Class A glassware (typically, the uncertainty at a specific temperature is written on the glassware) and syringes, precise room temperature, and the uncertainty inherent in all analytical balances despite calibration with National Institute of Standards and Technology (NIST) certified weights. The most challenging uncertainty to calculate is the uncertainty obtained from possible analyte to analyte and analyte to solvent interactions.

What Skills Are Needed to Create CRMs?
The first skill required when making a stock standard from a neat material is knowing which solvent is miscible or soluble with the analyte and if the analyte and solvent will quickly interact under the given storage conditions. If your working standard will be in a solvent different from your stock solvent, then the stock’s solvent is sometimes known as an intermediate. For example, if you want to spike used motor oil into water, you know that oil and water aren’t miscible. If you first dilute your oil with isopropanol at a reasonable concentration, the oil–isopropanol solution can be successfully spiked into the water matrix and you will have the used motor oil in solution with your water. The isopropanol is your intermediate solvent.

You have created all of your individual stock standards and now you want to combine portions of each stock to make

The Benefits of CRMs
CRMs, as opposed to RMs, give labs a competitive advantage and peace-of-mind. ISO 17025 recommends that accredited labs use CRMs whenever available. As a laboratory, you can trust that everything has been done to provide you with a high quality standard that helps to ensure that your data can be generated correctly the first time, and every time. After all, the quality of your data can directly affect consumer safety. Because the use of CRMs helps to ensure data quality, your services are of greater value to your clients and prospective clients.
your working standard mixture. Again, you need to ask, are the solvents miscible and will my analytes stay in solution? Will that solution be homogeneous? Will the analytes quickly interact with each other or with the solvents? To answer these questions you need experience. If you have done this before you will know what is going to happen. If this is your first time making this mixture, you won’t know for sure until you have finished the mixture and some time has passed. I will give you a hint: All organic chemicals interact as do many metals. To minimize your interaction and increase the shelf life, you need to know the correct storage conditions (colder is not always better) and which preservative, and how much of it, to add to your mixture. Storing a standard at too low of a temperature can negatively affect homogeneity and possibly cause some analytes to precipitate out of solution. It’s usually a good idea to sonicate or shake mixtures at room temperature before using, especially if they are cloudy or if you see solids floating in the solvent.

Many of you are probably now asking yourselves, what is a preservative? Analyte–solvent interaction can impact the pH of your solution and redox potential over time. Acetonitrile can break down into acetamide as the solvent ages, which makes your solvent basic. Urea-based pesticides will degrade under these conditions. A small amount of dilute formic acid can be added to your mixture to counter these reactions. Over time, halogenated hydrocarbons will interact with methanol to form acids. Low pH will degrade linear ketones very quickly. A dilute ammonium hydroxide solution will slow these reactions. Now you know why you should keep your acid sensitive and base sensitive analytes in separate mixtures. These are just a couple of examples of analyte–solvent interactions. There are hundreds of known interactions and many interactions are dependent on the number and concentration of your analytes in a mixture. Many laboratories request CRMs with all possible analytes at a high concentration in one ampule for cost savings. When your CRM expires in a couple of months, are you saving money? More organic compounds at higher concentrations mean there are greater chances for fast chemical interactions to occur. Use a stock product or, if a custom one is required, keep the concentrations reasonable and allow the manufacturer the option of separating analytes into multiple mixtures. The analytes’ shelf life will be longer and your headaches fewer.

How to Ensure Stability
Precision and accuracy are not the only concerns you might have about your standards; stability is also a major factor. The accidental use of a standard that has degraded will result in costly reanalysis and increased turn-around time, severely hurting your bottom line and your laboratory’s credibility. CRM manufacturers follow a set of ISO 17034 protocols to estimate a products shelf life. There are three types of data that can be used to determine a product’s shelf life: historical data, classical method data, and the accelerated method data. Historical data rely on the manufacturer’s knowledge and data of the exact reference material being made to know how long that material can be expected to remain accurate under the assigned storage conditions. Using the classical method, the manufacturer makes the product and tests retained samples of the standard over time until the product fails. As you can imagine, this method is very time consuming and I doubt that you want to wait years to find out when your standard will fail. Since most laboratories prefer custom standards, the only practical way to determine shelf life is by using the accelerated method. The accelerated method makes the assumption that the product failure (rate of degradation) increases as environmental conditions become harsher compared to the stated storage conditions. This ensures that the certified values for the analytes are within the stated uncertainties for the specified shelf life. The method is typically performed by heat stressing the standard. Several ampules of the standard are made and one is placed in a storage unit at the correct temperature. The other ampules are placed in heated environments at three or more temperatures between the stated storage temperature and 100 °C for a set time. The ampulized standards are analyzed sequentially using a metrologically validated method. If recovery of any of the analytes demonstrates a recovery of 95% or less, the standard is considered failed. This data is entered into an Arrhenius plot to provide a conservative estimate of shelf life. As the product ages, retained samples can be pulled from storage and tested using the classical method to determine if the shelf life can be lengthened or if it needs to be shortened.

Summary
As you can see, a great deal of effort is put into determining the values printed on your COAs. Even so, there can be unknowable aspects in the science of chemistry and the unforeseen can occur, but you can be assured that the best available technology was used to certify your reference materials. To reduce the possibility of the unforeseen, I recommend resisting the urge to amend your custom or catalog mixtures unless truly necessary. Also, when in doubt, let the professionals do it. You will save yourself time, money, and headaches.

Don Shelly is the Food and Environmental Product Manager, North America for LGC Standards in Manchester, New Hampshire. Direct correspondence to: don.shelly@lgcgroup.com
Choosing a Laboratory Information Management System (LIMS) for the Cannabis Industry

LabVantage Cannabis boosts laboratory testing productivity while reducing total cost and risk.

LabVantage Cannabis is a purpose-built Laboratory Information Management System (LIMS) that offers unique benefits to the cannabis testing industry. Product features are configured to meet cannabis testing requirements, taking into account the varied regulatory landscape. LabVantage Cannabis streamlines laboratory operations by offloading manual data entry and providing an electronic repository that can be easily queried for fast access to results. Cannabis Science and Technology (CST) recently sat down with Marty Pittman, senior product manager, to discuss how LabVantage Cannabis benefits testing laboratories and options for its deployment.

CST: Why do cannabis testing laboratories need a Laboratory Information Management System (LIMS)?

Pittman: Initially, most laboratories find that the amount of data associated with their customers’ testing is minimal enough to be managed in spreadsheets and other paper formats. However, as their customers’ businesses grow, the amount of data that testing laboratories have to work with increases. A LIMS can resolve much of the extra workload, helping laboratories to focus more on testing and less on managing data.

CST: What is unique about the LabVantage Cannabis product, which is LabVantage Solutions’ LIMS for this industry?

Pittman: LabVantage Cannabis, which is derived from our core LabVantage product, is configured for the cannabis industry. For example, American Herbal Pharmacopoeia methods that are specific to cannabis testing are included. This saves customers a lot of time in setting up and configuring the LIMS before they can make a product. LabVantage also provides a portal that cannabis growers can use to request testing directly. This feature facilitates sample collection, testing, and return of results. In addition, LabVantage provides an interface with government systems, keeping regulatory monitoring of the cannabis product and testing in one place.

CST: How do speed and cost of implementation of LabVantage Cannabis compare with that of a traditional LIMS deployment?

Pittman: When implementing a LIMS out of the box, a laboratory must make many determinations. These may include deciding upon what master data must be collected to comply with regulatory requirements, process flows in the laboratory, what data must be gathered from which instruments, and the data approval process. LabVantage Solutions worked with its customers in the cannabis industry to identify these items upfront. As a result, LabVantage
Cannabis incorporates much of the information that would otherwise have to be discovered, documented, and configured from scratch. This allows the LabVantage Cannabis customer to jump right in and begin using the LIMS.

**CST: What features make LabVantage Cannabis unique to cannabis testing?**

**Pittman:** In the cannabis industry, there are some unique requirements when requesting samples. The request does not come from an internal laboratory source; external growers or other cannabis product manufacturers must be able to request the test. LabVantage Cannabis provides a portal that allows the grower to submit the request for testing, which initiates the sample collection process. LabVantage also includes a chain of custody that is in place when the sample is received so that it is processed in a known workflow. Data are peer reviewed to ensure that there are no transcription errors. Finally, a Certificate of Analysis is generated.

During the process (from sample collection to completion of the Certificate of Analysis), data must be provided to state governments at certain points to track the cannabis material and testing results. As I mentioned, LabVantage provides the interface to do that. Much of the functionality that is needed for American Herbal Pharmacopoeia testing is provided upfront. In some cases, this is extended to allow for automated instrument integration. This enables an instrument to produce an output file, which can be parsed and imported into the LIMS. This spares an analyst from having to manually key data from the instruments into the LIMS for tracking and generation of the Certificate of Analysis.

**CST: How does LabVantage Cannabis address the varied regulatory landscape?**

**Pittman:** The highly flexible configuration options of our core LabVantage LIMS are retained in the LabVantage Cannabis product. Customers can modify LabVantage Cannabis as needed to meet their own requirements. For example, it could be configured on the basis of where the customer is located, what regulatory bodies they must comply with, or what laboratory methods they have in place.

**CST: Please describe the two options offered for the deployment of LabVantage Cannabis.**

**Pittman:** LabVantage has a software as a service (SaaS)-based option and a traditional enterprise option in which the customer purchases a license. The latter is typically used for implementations in on-premise hardware or in customer-owned cloud environments. The customer needs an annual subscription for maintenance, but owns the product. As such, they are responsible for providing all support and testing of the operating system, application server, and database server, as well as for working with LabVantage to support the application itself.

The SaaS model is a “pay-as-you-go” solution in which the customer pays an annual subscription fee per user. In this case, LabVantage manages the operating system, application server, and database server; the customer has to deal only with the LIMS itself. This allows the customer to focus on its laboratory processes (i.e., collecting and managing data) without having to worry about the behind-the-scenes hardware and operating systems needed to support the environment.

Some LabVantage customers have reported that the lower yearly cost of the SaaS model is an advantage. Other customers prefer the enterprise model because they can purchase it upfront and then just pay for the annual subscription for maintenance. Offering these two models gives customers a lot of flexibility in terms of deciding their payment schedule.

**CST: How does all of this benefit users?**

**Pittman:** The biggest benefit is that users can focus more on their specialty—namely, the testing of laboratory materials. By having the LIMS in place, users worry less about transcription errors in Excel spreadsheets and where papers are located in the laboratory. LabVantage gives laboratories the ability to store all of their data in a central location where it can be easily accessed when customers have questions about testing results or the status of samples. LabVantage offloads many of the day-to-day physical transactions of entering data or moving papers from desk to desk. Users can concentrate on the analysis of materials and tracking of results.

**CST: Lastly, when a customer invests in LabVantage Cannabis, how do they know if they are getting their money’s worth?**

**Pittman:** LabVantage Cannabis confers advantages in multiple areas. Customers quickly find that they can process more samples with the same number of resources. It allows chemists to spend more time on bench-top analysis and less on data entry. In addition, electronic data can be mined, queried, and analyzed. LabVantage Cannabis customers can drill down to look for trends in their data. The ability to perform more data analysis could also aid the cannabis grower, by identifying plant strains that are “good” versus those that may have undergone mutations.
Holding Data to a Higher Standard, Part II: When Every Peak Counts—A Practical Guide to Reducing Contamination and Eliminating Error in the Analytical Laboratory

Chemical reference standards have been an important component in accurate analysis for decades. Over the years, the challenges facing chemical laboratories have changed. As a manufacturer of certified reference materials (CRMs), many questions have arisen during our daily interaction with customers on how to best use CRMs. Sometimes a customer is not even aware that the issue they are questioning is really an problem of contamination or error. A common scenario is that a scientist will express a concern that their standard is too high in particular elements. Usually during the conversation, our scientists discover that the customer...
is inadvertently allowing contamination into their analysis. Contamination and error can occur at almost any point of the process and then can be magnified as the method and analysis runs its course. Modern instrumentation has raised the bar or in this case lowered the limits of detection to the parts-per-billion (ppb) or even parts-per-trillion (ppt) levels. The concept of such small measurements would have been almost inconceivable during the emergence of modern laboratory analysis.

To put this into a different context, 1 ppb expressed as a unit of time would be 1 s in 32 years and 1 ppt would be 1 s in 320 centuries! In the past, issues of laboratory contamination were problematic but now contaminants, even in trace amounts, can severely alter results. It is hard to imagine that such small amounts of contamination can dramatically change laboratory values.

Most questions about contamination come in the form of an inquiry about a particularly high result for some common contaminant. In most cases, the root of that contamination can be traced to a common source. The most common sources of standard and sample contamination are found in the laboratory: reagents, labware, laboratory environment, storage, and personnel.

**Water Quality**

Water is one of the most basic yet most essential laboratory components. Most scientists are aware that the common perception that all water is the same is untrue. There are many types, grades, and intended uses for water. Water is most often used in two ways in the laboratory: as a cleaning solution and as a transfer solution for volumetric or gravimetric calibrations or dilutions. In both of these uses, the water must be clean to reduce contamination and introduce error into the process. Poor quality water can cause a host of problems from creating deposits in labware or inadvertently increasing a target element or analyte concentration in solution.

The confusion starts when laboratories are unsure about which type of water they get from their water filtration system. ASTM has guidelines that designate different grades of water. Table I shows parameters for the four ASTM types of water (1).

The actual type of water produced by a commercial laboratory water filtration system can vary in pH, solutes, and soluble silica. Critical analytical processes should always require a minimum quality of ASTM Type I
water. All trace analysis standards, dilutions, dissolutions, extractions, and digestions should be conducted with the highest purity of water. Analysts who use CRMs and perform quantitative analysis need to use quality water so as not to contaminate their CRMs, standards, and samples with poor quality water.

High-purity water is often achieved in several stages in multiple processes that remove physically and chemically potential contaminating substances. Municipal water supplies often test their own water sources on an annual basis, but that does not mean it is applicable for use in laboratory applications. Municipal water can become contaminated from its distribution point, especially when left static sitting in pipes, tubing, and hoses. Water left stationary in a laboratory water system can be exposed to leaching of elements and compounds from the piping and hoses. In one experiment conducted at SPEX CertiPrep, leaching solutions of either ASTM Grade I water or 5% nitric acid in ASTM Grade I water were run through lengths of silicon and neoprene laboratory tubing. The solutions were collected and tested by inductively coupled plasma–mass spectrometry (ICP-MS). Many common elements were found to have leached into the solutions from exposure to the laboratory tubing adding potential contamination to critical analytical processes, which can be seen in Table II. If the tubing was used to transfer acidified solutions between vessels the acid further increased the contamination, elements such as lead increased from 0.1 μg/L in the ASTM I deionized (DI) water to 3 μg/L in the silicon tubing with 5% nitric acid and 2 μg/L in the neoprene tubing with 5% nitric acid.

Another potential source of contamination of laboratory water is phthalates. Phthalates are ubiquitous in the environment and in the

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**Table I:** ASTM designations for reagent laboratory water (1)

<table>
<thead>
<tr>
<th>Requirement</th>
<th>ASTM Type</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>I</td>
</tr>
<tr>
<td>Use</td>
<td>Critical laboratory applications and processes</td>
</tr>
<tr>
<td>Specific Resistance (megohm/m) (max)</td>
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<tr>
<td>pH</td>
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</tr>
<tr>
<td>Sodium (max)</td>
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</tr>
<tr>
<td>Total Silica (max)</td>
<td>3 μg/L</td>
</tr>
<tr>
<td>Total Organic Carbon (max)</td>
<td>50 μg/L</td>
</tr>
</tbody>
</table>

**Table II:** Elemental leaching from laboratory tubing (μg/L) (2)

<table>
<thead>
<tr>
<th>Tubing Type</th>
<th>Silicon</th>
<th>Neoprene</th>
<th>Silicon</th>
<th>Neoprene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Leaching Solution</td>
<td>DI H2O</td>
<td>DI H2O</td>
<td>5% HNO3</td>
<td>5% HNO3</td>
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<tr>
<td>Fe</td>
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<td>0</td>
<td>27</td>
<td>5</td>
</tr>
<tr>
<td>K</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Mg</td>
<td>7</td>
<td>0.5</td>
<td>8</td>
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<td>0</td>
</tr>
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</tr>
<tr>
<td>Zn</td>
<td>1</td>
<td>55</td>
<td>4</td>
<td>50</td>
</tr>
</tbody>
</table>

**Figure 1:** Phthalate concentrations in laboratory water sources (ppb) (3).
laboratory. Plastics are found everywhere from bottles, tubing, caps, and containers. In a laboratory water study performed at SPEX CertiPrep, we examined bisphenol A and phthalate content present in a variety of laboratory water sources including bottled high performance liquid chromatography (HPLC)-grade, liquid chromatography–mass spectrometry (LC–MS)-grade water, municipal tap water, and water obtained from our in-house DI (ASTM II) water source (3). The in-house DI water was tested from a carboy filled within the laboratory, and directly from the faucet outlet. The faucet samples were collected after sitting overnight and a second sample was collected after the system had been flushed with multiple gallons of fresh water. Phthalates were found in all the water sources with the highest total phthalates found in the HPLC bottled water with about 91 ppb of phthalates as seen in Figure 1.

To reduce contamination from laboratory water sources, the first line of attack is to choose the correct water source for the given application. It is also important to realize that water can change quality over time depending on the storage conditions. Bottled-water sources can leach organic and inorganic contaminants from the bottle, cap, and liners. Bottled water can also have an expiration date and a shelf life that should be checked before use. Water that has been decanted into other vessels can be exposed to many types of contaminants including dust, microbial growth, and oxidation effects. Water left in liquid systems such as HPLC, LC–MS, or ion chromatography (IC) systems should be changed frequently to prevent contamination and microbial growth.

### Reagents

An analytical laboratory often uses a large amount of various reagents, solvents, and acids of varying quality and contamination levels. These chemical components can be a large economic investment for a laboratory, but also a large source of potential contamination. Just as in the case of water, there are different types or grades of chemicals, reagents, acids, and solvents. Some designations are set forth by standards set by the U.S. Pharmacopeia (USP) or the American Chemical Society (ACS). Other types or grades of material are designated by individual manufacturers based on intended use. Some are general laboratory grades with intended use for noncritical applications while other grades usually high in purity and low in contaminants are designated for more-critical analyses. Table III shows some general descriptions of types and grades of solvents and their intended uses.

Many analytical laboratories use a variety of chromatography and spectrometry instruments such as gas chromatography (GC), GC–MS, HPLC, and LC–MS in their analyses. Solvents play a large role in these analytical techniques as either mobile phases or matrices for analysis. During sample preparation, many laboratories expose samples to a variety of solvents ahead of introduction to an analytical instrument. In some cases, the kind of solvent is chosen to best fit the technique. LC, GC, and MS systems each have different modes of analysis that benefit from specific chemical or physical properties found in solvents. For example, in GC and GC–MS, the most widely used solvents have low boiling points, are eluted quickly, and don’t interfere with the target analytes. Solvents with boiling points

| Table III: Grades of laboratory solvents and their uses |
|----------------|----------------|----------------|
| **Grade**      | **Application or Use** | **Analytical** |
| ACS            | General procedures  | Meets or exceeds ACS specifications |
| Anhydrous      | Water-sensitive reactions and synthesis | Low water levels (10–30 ppm) |
| Biotechnical   | Biotechnical applications  | Low water, low residue, low UV |
| Environmental  | Environmental analysis, HPLC, trace organic | Trace organics |
| Food/FCC grade | Food and drug applications | Meets specifications of Food Chemicals Codex (FCC) |
| GC             | GC applications      | ppb levels of contamination |
| HPLC           | HPLC applications    | Sub-micrometer filtration, some low UV absorbance |
| LC–MS          | LC–MS applications   | Low ionic impurities, <0.1 ppm |
| Pesticide residue | Pesticide, environmental analyses, trace analyses, and GC with various detectors | Meets or exceeds ACS pesticide specifications |
| Reagent        | General laboratory use | >95% purity |
| Spectrophotometric | UV applications | UV, vis, IR |
| Technical      | General laboratory use | Noncritical tasks |
| USP            | Food and drug applications | Meets or exceeds USP specifications |
within the analytical range of the target analytes have the potential to be coeluted with the targets and hinder quantitation. (See Table IV.)

LC and MS techniques depend on the type of analysis being performed. Normal-phase LC uses polar columns and nonpolar solvents such as hexane and cyclohexane. Reversed-phase LC uses nonpolar columns such as octadecyl (C18) columns with polar solvents including methanol, water, and acetonitrile. If ultraviolet–visible light diode-array detection (UV-vis DAD) or equivalent is used then the wavelength of the solvent also becomes important. The wavelength of the mobile phase and solvents used should be outside of the wavelengths of the target analytes in the analysis. The most common range for a typical reverse phase is above 190 nm UV cutoff for acetonitrile and below 300 nm, which is the UV cutoff for acetone, both popular solvents for LC applications seen in Table IV.

Solvents are both a material that can become contaminated and a source of contamination. Solvents can be contaminated by particles such as dust, rust, and mold. The solvents can also be contaminated by gases, oxidation, or compounds like the phthalates found in seals and bottle closures. Some solvents have added preservatives that could add contaminants to analysis or leach additional elements from the storage containers.

There are many persistent solvents that can be found in the laboratory which can cross-contaminate samples by their presence. Some persistent solvents include dichloromethane which can cause chlorine contamination as well as dimethyl sulfoxide (DMSO) and carbon disulfide, which can add sulfur residues. There are solvents that react with air to form peroxides, which can cause contamination and safety issues in the laboratory. Acids are another laboratory reagent that can become both a potential danger and a potential contaminant. Acids by their nature are oxidizers and many of the strongest acids are used in the processing of samples for inorganic analysis. Common acids in digestion and dissolution include perchloric acid, hydrofluoric acid, sulfuric acid, hydrochloric acid, and nitric acid. Many of these acids are commercially available in several grades from general laboratory or reagent grade to high-purity trace metal–grade acids. Acid grades often reflect the number of sub-boiling distillations the acid undergoes for purification before bottling. The more an acid is distilled, the higher the purity. These high-purity acids have the lowest amount of elemental contamination but can become very costly at up to 10 times the cost of the reagent grade of acids.

Often the question is asked if high-purity acids are necessary in sample preparation if the laboratory is using a high-quality ICP-MS-grade CRM. Clean acids used in sample preparation, digestion, and preservation can be very costly. But, the difference between the amounts of contamination in a low-purity acid and a high-purity acid can be dramatic. High-quality standards for use in parts-per-billion and parts-per-trillion analyses use the highest purity acids available to reduce all possible contamination from the acid source. An example of potential contamination is an aliquot of 5 mL of acid containing 100 ppb of Ni as contaminant, used for diluting a sample to 100 mL, can introduce 5 ppb of Ni into the sample.

To reduce contamination it is recommended that high-purity acids be used to dilute and prepare standards and samples when possible. In addition to using pure acid, it is important that the chemist check the acid’s certificate of analysis to identify the elemental contamination levels present in the acid. Some laboratories prefer to use blank subtractions to negate the background contamination, but blank subtraction for acids can only work in a range well over the instrumental level of detection. If blank subtraction causes an analytical result to fall below the instrument’s level of detection, it should not be used.

<table>
<thead>
<tr>
<th>Solvent</th>
<th>MW</th>
<th>UV Cutoff</th>
<th>BP (°C)</th>
<th>Polarity Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>18</td>
<td>NA</td>
<td>100</td>
<td>10.2</td>
</tr>
<tr>
<td>Methanol</td>
<td>32</td>
<td>205</td>
<td>65</td>
<td>5.1</td>
</tr>
<tr>
<td>Acetonitrile</td>
<td>41.1</td>
<td>190</td>
<td>82</td>
<td>5.8</td>
</tr>
<tr>
<td>Acetone</td>
<td>58.1</td>
<td>330</td>
<td>56</td>
<td>5.1</td>
</tr>
<tr>
<td>2-Propanol</td>
<td>60.1</td>
<td>205</td>
<td>82</td>
<td>3.9</td>
</tr>
<tr>
<td>Tetrahydrofuran</td>
<td>72.1</td>
<td>210</td>
<td>66</td>
<td>4</td>
</tr>
<tr>
<td>Ethyl ether</td>
<td>74.1</td>
<td>218</td>
<td>35</td>
<td>2.8</td>
</tr>
<tr>
<td>DMSO</td>
<td>78.1</td>
<td>262</td>
<td>189</td>
<td>7.2</td>
</tr>
<tr>
<td>Cyclohexane</td>
<td>84.2</td>
<td>202</td>
<td>81</td>
<td>0.2</td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>84.9</td>
<td>233</td>
<td>40</td>
<td>3.1</td>
</tr>
<tr>
<td>Hexane</td>
<td>86.2</td>
<td>195</td>
<td>69</td>
<td>0.1</td>
</tr>
<tr>
<td>Ethyl acetate</td>
<td>88.1</td>
<td>255</td>
<td>77</td>
<td>4.4</td>
</tr>
<tr>
<td>Toluene</td>
<td>92.1</td>
<td>285</td>
<td>111</td>
<td>2.4</td>
</tr>
<tr>
<td>Isooctane</td>
<td>114</td>
<td>205</td>
<td>99</td>
<td>0.1</td>
</tr>
</tbody>
</table>
Volumetrics and Labware

Volumetric measurement is a common repeated daily activity in most analytical laboratories. Many processes in the laboratory from sample preparation to standards calculation depend on accurate and contamination-free volumetric measurements. Unfortunately, laboratory volumetric labware, syringes, and pipettes are among the most common sources of contamination, carryover, and error in the laboratory.

### Table V: Dispensing volume error associated with various size syringes (4)

<table>
<thead>
<tr>
<th>Syringe Size</th>
<th>Volume Dispensed (μL)</th>
<th>% of Syringe Volume</th>
<th>% Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>2</td>
<td>20%</td>
<td>23.15%</td>
</tr>
<tr>
<td>10</td>
<td>5</td>
<td>50%</td>
<td>8.16%</td>
</tr>
<tr>
<td>10</td>
<td>10</td>
<td>100%</td>
<td>2.72%</td>
</tr>
<tr>
<td>25</td>
<td>2</td>
<td>8%</td>
<td>8.82%</td>
</tr>
<tr>
<td>25</td>
<td>5</td>
<td>20%</td>
<td>5.47%</td>
</tr>
<tr>
<td>25</td>
<td>10</td>
<td>40%</td>
<td>2.37%</td>
</tr>
<tr>
<td>25</td>
<td>20</td>
<td>80%</td>
<td>1.05%</td>
</tr>
<tr>
<td>25</td>
<td>25</td>
<td>100%</td>
<td>1.25%</td>
</tr>
<tr>
<td>100</td>
<td>10</td>
<td>10%</td>
<td>6.09%</td>
</tr>
<tr>
<td>100</td>
<td>25</td>
<td>25%</td>
<td>1.67%</td>
</tr>
<tr>
<td>100</td>
<td>50</td>
<td>50%</td>
<td>0.64%</td>
</tr>
<tr>
<td>100</td>
<td>100</td>
<td>100%</td>
<td>0.61%</td>
</tr>
<tr>
<td>1000</td>
<td>250</td>
<td>25%</td>
<td>1.05%</td>
</tr>
<tr>
<td>1000</td>
<td>500</td>
<td>50%</td>
<td>1.14%</td>
</tr>
<tr>
<td>1000</td>
<td>1000</td>
<td>100%</td>
<td>0.47%</td>
</tr>
</tbody>
</table>

### Table VI: Compound carryover found in syringe washes (ppm of carryover) (4)

<table>
<thead>
<tr>
<th>Syringe Wash Number</th>
<th>1000 μL</th>
<th>10 μL</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>24.4</td>
<td>360.51</td>
</tr>
<tr>
<td>2</td>
<td>0.69</td>
<td>46.96</td>
</tr>
<tr>
<td>3</td>
<td>0.06</td>
<td>8.12</td>
</tr>
<tr>
<td>4</td>
<td>0.03</td>
<td>4.1</td>
</tr>
<tr>
<td>5</td>
<td>0.04</td>
<td>2.02</td>
</tr>
<tr>
<td>7</td>
<td>0.01</td>
<td>1.37</td>
</tr>
<tr>
<td>10</td>
<td>0.03</td>
<td>1.37</td>
</tr>
<tr>
<td>15</td>
<td>0.02</td>
<td>1.13</td>
</tr>
<tr>
<td>20</td>
<td>0.01</td>
<td>0.43</td>
</tr>
</tbody>
</table>

*Introducing a newly updated California mixture - now including most recent pesticides.
The root of these errors is based on the four “I” errors of volumetrics:

- Improper use
- Incorrect choice
- Inadequate cleaning
- Infrequent calibration.

These four “I”s can lead to error and contamination, which negate all intent of careful measurement processes.

The first two “I”s stand for improper use, meaning that the volumetric is not used correctly or the incorrect choice is made. Many errors can be avoided by understanding the markings displayed on the volumetrics and choosing the proper tool for the job. There is a lot of information displayed on volumetric labware. Most labware, especially glassware, is designated as either Class A or Class B labware. Class A glassware is a higher quality analytical class of glassware whereas Class B glassware is a lower quality glassware with a larger uncertainty and tolerance. If a critical measurement process is needed, then only Class A glassware should be used for measurement.

Other information that can be found on labware is the name of the manufacturer, country of origin, tolerance or uncertainty of the measurement of the labware, and a series of descriptors that indicate how the glassware should be used. Labware can be marked with letters that designate the purpose of the container. If a volumetric is designed to contain liquid it will be marked by either the letters TC or IN. Labware that is designated to deliver liquid will be marked by either the letters TD or EX. Sometimes there are additional designations such as wait time or delivery time inscribed on the labware. The delivery time refers to a period of time required for the meniscus to flow from the upper volume mark to reach the lower volume mark. The wait time refers to the time needed for the meniscus to come to rest after the residual liquid has finished flowing down from the wall of the pipette or vessel.

A second type of improper use and incorrect choice can be seen in the selection of pipettes and syringes for analytical measurements. Many syringe manufacturers recommend a minimum dispensing volume of approximately 10% of the total volume of the syringe or pipette. A study by SPEX CertiPrep showed that dispensing such a small percentage of the syringe’s total volume created a large amount of error. In this study, four syringes, 10 μL, 25 μL, 100 μL, and 1000 μL were used to dispense between 8–100% of the syringe’s total volume of water. Each volume was weighed and replicated 10 times by several analysts and the results were averaged together to calculate average error.

The largest rates of error were seen in the smaller syringes of 10 and 25 μL. Dispensing 20% of the 10 μL syringe created 23% error. Error only dropped down to below 5% as the volume dispensed approached 100%. In the larger syringes, measurements over 25% were able to see error in and around 1%. The larger syringes were able to get closer to the 10% manufacturer’s dispensing minimum without a large amount of error, but the error did drop as the dispensed volume approached 100%, which is seen in Table V.

The third “I” of volumetric error is inadequate cleaning. Many volumetrics can be subject to memory

### Table VII: Major elemental impurities found in laboratory container materials (2)

<table>
<thead>
<tr>
<th>Material</th>
<th>Number of Elements</th>
<th>Total ppm</th>
<th>Major Impurities</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polystyrene-PS</td>
<td>8</td>
<td>4</td>
<td>Na, Ti, Al</td>
</tr>
<tr>
<td>Tetrafluoroethylene (TFE)</td>
<td>24</td>
<td>19</td>
<td>Ca, Pb, Fe, Cu</td>
</tr>
<tr>
<td>Low density PE-LDPE</td>
<td>18</td>
<td>23</td>
<td>Ca, Cl, K, Ti, Zn</td>
</tr>
<tr>
<td>Polycarbonate-PC</td>
<td>10</td>
<td>85</td>
<td>Cl, Br, Al</td>
</tr>
<tr>
<td>Polymethyl pentene-PMP</td>
<td>14</td>
<td>178</td>
<td>Ca, Mg, Zn</td>
</tr>
<tr>
<td>Fluorinated ethylene propylene (FEP)</td>
<td>25</td>
<td>241</td>
<td>K, Ca, Mg</td>
</tr>
<tr>
<td>Borosilicate glass</td>
<td>14</td>
<td>497</td>
<td>Si, B, Na</td>
</tr>
<tr>
<td>Polypropylene-PP</td>
<td>21</td>
<td>519</td>
<td>Cl, Mg, Ca</td>
</tr>
<tr>
<td>High density PE-HDPE</td>
<td>22</td>
<td>654</td>
<td>Ca, Zn, Si</td>
</tr>
</tbody>
</table>

### Table VIII: Elemental impurities found in nitric acid distilled in clean laboratories versus regular laboratories (2)

<table>
<thead>
<tr>
<th>Element</th>
<th>Regular Laboratory</th>
<th>Clean Laboratory</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>60</td>
<td>15</td>
</tr>
<tr>
<td>As</td>
<td>0.17</td>
<td>&lt;0.02</td>
</tr>
<tr>
<td>Ca</td>
<td>150</td>
<td>100</td>
</tr>
<tr>
<td>Cd</td>
<td>0.3</td>
<td>0.003</td>
</tr>
<tr>
<td>Cr</td>
<td>2.5</td>
<td>0.4</td>
</tr>
<tr>
<td>Cu</td>
<td>1.7</td>
<td>0.23</td>
</tr>
<tr>
<td>Fe</td>
<td>50</td>
<td>9</td>
</tr>
<tr>
<td>Mg</td>
<td>10</td>
<td>4</td>
</tr>
<tr>
<td>Mn</td>
<td>1.1</td>
<td>0.1</td>
</tr>
<tr>
<td>Mo</td>
<td>0.8</td>
<td>0.03</td>
</tr>
<tr>
<td>Pb</td>
<td>0.5</td>
<td>0.4</td>
</tr>
<tr>
<td>Sb</td>
<td>0.04</td>
<td>0.013</td>
</tr>
<tr>
<td>Zn</td>
<td>5.5</td>
<td>0.7</td>
</tr>
</tbody>
</table>
effects and carryover. In critical laboratory experiments, labware sometimes needs to be separated by purpose and use. Labware subject to high levels of organic compounds or persistent inorganic compounds can develop chemical interactions and memory effects. It is also sometimes difficult to eliminate carryover from labware and syringes even when using a manufacturer’s stated instructions. For example, many syringes are cleaned by several repeated solvent rinses before use. A study of syringe carryover by SPEX CertiPrep showed that some syringes are subject to high levels of chemical carryover despite repeated rinses.

In this study, several syringes ranging in volume from 10 μL to 1000 μL were used to dispense a 2000 μg/mL internal standard mix of deuterated compounds. The subsequent washes were collected and tested by GC–MS to determine the amount of carryover in each wash (see Table VI).

The larger syringes needed less rinses to reduce carryover than the smaller 10-μL syringes. The smaller syringes had more than 1 ppm carryover through over 15 rinses. The typical number of rinses usually employed to rinse syringes is between three and five, which in the case of the smaller syringe would not be adequate to clear all the carryover from the syringe.

The final source of error is infrequent calibration. Many laboratories have schedules of maintenance for equipment such as balances and automatic pipettes, but often overlook calibration of reusable burettes, pipettes, syringes, and labware. Under most normal use, labware often does not need frequent calibration but there are some instances where a schedule of recalibration should be used. Any glassware or labware in continuous use for years should be checked for calibration. Glass manufacturers suggest that any glassware used or cleaned at high temperatures, used for corrosive chemicals, or autoclaved should be recalibrated more frequently.

It is also suggested that under normal conditions that soda-lime glass be checked or recalibrated every five years and borosilicate glass after it has been in use for 10 years. The error associated with the use of volumetrics can be greatly reduced by choosing the correct volumetric for the task, using the tool properly, and by making sure the volumetrics are properly cleaned and calibrated before use.

Inorganic analysts know that glassware is a source of contamination. Even clean glassware can contaminate samples with elements such as boron, silicon, and sodium. If glassware, such as pipettes and beakers, is reused, the potential for contamination escalates. A study was made of residual contamination at SPEX CertiPrep of our pipettes after they were manually and automatically cleaned using a pipette washer (2).

An aliquot of 5% nitric acid was drawn through a 5-mL pipette after the pipette was manually cleaned according to standard procedures. The aliquots were analyzed by ICP-MS. The results showed that significant residual contamination still persisted in the pipettes despite a thorough manual cleaning procedure.

The experiment was repeated using a pipette washer especially made for use in parts-per-trillion analysis. The pipette washer repeatedly forced deionized water through the pipettes for a set time period. The pipettes were cleaned in the pipette washer, and then the same aliquot of 5% nitric acid was drawn through the 5-mL pipettes. The aliquot was analyzed by ICP-MS. The automated washer reduced the contamination significantly over manual cleaning of the pipettes. The reduction of contamination by moving from manual cleaning to an automated cleaning process was clear. High levels of contamination of sodium and calcium (almost 20 ppb) dropped to <0.01 ppb. Other common contaminants including lead and iron dropped from 5.4 and 1.6 ppb, respectively, to less than 0.01 ppb.

The reduction of contamination in labware can depend on the material of the labware and its use. Different materials contain many types of elemental and organic potential contamination as seen in Table VII (5). Trace inorganic analyses are best performed in polymer or high purity quartz vessels, such as fluorinated ethylene propylene (FEP), and minimize contact with borosilicate glass. Metals such as Pb and Cr are highly absorbed by glass but not by plastics. On the other hand, samples containing low levels of Hg (parts-per-billion levels) must be stored in glass or fluoropolymer because Hg vapors diffuse through polyethylene bottles.

**Laboratory Environment and Personnel**

All laboratories believe they observe a level of laboratory cleanliness. Most chemists recognize that there are inherent levels of contamination present in all laboratories. A common
belief is that the small amounts of environmental and laboratory contamination cannot truly change the analytical results. To test the background level of contamination in a typical laboratory, samples of nitric acid were distilled in both a regular laboratory and in a clean-room laboratory with special air handling systems (HEPA filters). The nitric acid distilled in the regular laboratory had high amounts of aluminum, calcium, iron, sodium, and magnesium contamination. Table VIII shows that the acid distilled in the clean room had significantly lower amounts of most contaminants (2).

Laboratory air also can contribute to contamination of samples and standards. Common sources of air and particulate matter contamination are from surfaces and building materials such as ceiling tiles, paints, cement, and dry wall. Surface contaminants can be found in dust and rust on shelves, equipment, and furnature. Dust contains many different Earth elements such as sodium, calcium, magnesium, manganese, silicon, aluminum, and titanium. Dust can also contain elements of human activities (Ni, Pb, Zn, Cu, As) and organic compounds like pesticides, persistent organic pollutants (POP), and phthalates. The dust and rust particles can contaminate open containers in the laboratory or enter containers by charge transfer from friction by the triboelectric effect. The triboelectric effect or triboelectric charging is when materials become charged after coming into contact with a second material creating friction. The most common example of this effect is seen when hair sticks to a plastic comb after a static charge is created.

The polarity and the strength of the electrical charge is dependent upon the type of material and other physical characteristics. Many materials in the laboratory have strong positive or negative triboelectric charges as shown in Figure 2. In the laboratory, materials like dust, air, skin, and lead have extreme positive charges and can be attracted to the strongly negative charge of PTFE or other plastic bottles when the bottle is opened and friction is created, inducing a charge.

Laboratory personnel can add their own contamination from laboratory coats, makeup, perfume, and jewelry. Aluminum contamination can come from laboratory glassware, cosmetics, and jewelry. Many other common elements can be brought in as contamination from lotions, dyes, and cosmetics. Even sweat and hair can cause elevated levels of sodium, calcium, potassium, lead, magnesium, and many ions. If a laboratory is seeing an unusually high level of cadmium in the samples it could be from cigarettes, pigments, or batteries. If the levels of lead are out of range, contamination can be from paint, cosmetics, and hair dyes. Figure 3 shows potential sources of common elemental contamination from outside products.

Laboratory environment and personnel contamination can be reduced by limiting use of personal care products, jewelry, and cosmetics that could contain contamination and interfere with critical analyses. Laboratory coats can collect all types of contamination and should only be worn in the laboratory to avoid cross contamination from other laboratories and the outside world. The laboratory surfaces should be kept clean. Deionized water can be used to wipe down work surfaces. Laboratory humidity can be kept above 50% to reduce static charge. An ethanol- or methanol-soaked laboratory wipe can be used to reduce static electricity as it evaporates.

Even with clean laboratory practices in place, erroneous results can often find their way into sample analysis. To eliminate some of these spurious results, replication of blanks and sample dilutions can be used. The blank results should be averaged and the sample run values can either be minimally selected or averaged. The difference between the two values can then be plotted against a curve established against two more standards. A minimum of two standard points can be used if the chance of contamination is minimal, such as in the case of rare or uncommon elements. Additional standard points should be considered if the potential for contamination is high with common elements such as aluminum, sodium, and magnesium. Multiple
aliquots of blanks and dilutions can also be used to further minimize analytical uncertainty.

**General Principles and Practices**

Labs should follow a general regime of three runs each of wash-rinse runs, blank runs, and sample runs, as well as single runs of sample plus spike, and standard or spike runs without sample to use as a control solution to evaluate recovery.

Analysts must realize that the cleanliness and accuracy of their procedures, equipment, and dilutions affect the quality of the standards and samples. Many laboratories will dilute CRMs to use across an array of procedures and techniques. This in-house dilution of CRMs can be a savings to the laboratory but in the final analysis can be a source of error and contamination.

CRM manufacturers design standards for particular instruments to obtain the highest level of accuracy and performance for that technique. They also use calibrated balances, glassware, and instruments to ensure the most accurate standards are delivered to customers. Certifications such as ISO 9000, ISO 17025, and 17034 assure customers that procedures are being followed to ensure quality and accuracy in those standards. After those CRMs are in chemists’ hands, it is then their responsibility to use all possible practices to keep their analysis process free from contamination and error.

**References**


**It looks pure and clear...**

**Looks can be deceiving.**

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Most of the traditional methodologies for the determination of cannabinoids are based on solvent extraction, filtration, and concentration. These techniques are cumbersome, time-consuming, and suffer from analyst-to-analyst variability while producing data of limited value. Many laboratories routinely “screen” each sample to quickly determine the potential for matrix interference and instrument contamination while providing an estimate of the target compound’s concentration. A good “screening” method is simple (that is, minimal or no sample preparation), fast (analysis in less than 5 min), and semiquantitative. This article describes how thermal desorption (TD)-gas chromatography–mass spectrometry (GC–MS) analysis eliminates conventional sample preparation regimes and can be used as a good rapid screening technique.

Rojin Belganeh and William Pipkin
analysis (EGA), starts with the acquisition of a thermal profile (that is, detector response as a function of sample temperature) of each sample type. To perform EGA, a short, deactivated tube (2.5 m, 0.15 mm i.d.) is used to connect the injection port to the MS detector. The sample is dropped into the furnace at a relatively low temperature (40–100 °C). The furnace is then programmed to a much higher temperature (600–800 °C). Compounds “evolve” from the sample as the temperature increases. A plot of detector response versus furnace temperature is obtained. Extracted ion chromatograms (EIC) are used to identify the thermal zone over which specific compounds of interest evolve from the sample. Now, these optimum TD temperatures can be used in subsequent TD-GC–MS experiments to introduce the key components of interest while minimizing introduction of the matrix. Only this portion of the sample is actually transferred (that is, injected on) to the analytical column. Injecting only a small portion of the sample provides immediate benefits to the laboratory, such as:

- The high boiling fraction of the sample remains in the sample cup. This eliminates the need for a high-temperature bake out. Thus, column lifetime increases, there is little to no system contamination, and run-to-run cycle time decreases.
- More sample can be put in the sample cup, which has the effect of lowering detection limits—without affecting instrument performance or cycle time.

With respect to the analysis of cannabinoids, it is important to keep in mind that TD-GC–MS is based on the volatilization of target compounds from the matrix. Those compounds that are thermally labile or easily converted to an alternative compound need to be identified. In these instances, it is the reformed compounds that are identified and monitored. Decarboxylation is forced to completion which could give the “screening determinations” higher values: the concentration range increases and dilution factors are more accurately determined.

**Experimental**

Three edible samples were analyzed using the TD-GC–MS method for quantification of cannabinoids. The experiments were performed by a Frontier Multi-Mode Pyrolyzer (EGA/PY 3030D) directly interfaced to a benchtop GC–MS system. To automate the sequence and reduce the workload by increasing reliability of analysis, the Auto-Shot Sampler (AS-1020E) was combined with the multimode pyrolyzer. The vent-free
GC–MS adaptor that allows switching of a GC separation column without breaking vacuum on the MS detector was also used. The adaptor was utilized for switching the EGA tube to the separation column and vice versa without venting the MS detector, which saves time and increases productivity.

**Sample 1**

A commercial package of cannabis-infused chocolate brownie containing 10 brownie bites with the total of 100 mg tetrahydrocannabinol (THC) was used. According to the product label, each brownie bite contains 10 mg of THC. In this experiment, one of the brownie bites from the package was placed on an analytical balance, and the weight was recorded as 10.17 g. So, based on the label, there is 10 mg/10 g = 1 mg/g of THC present in that brownie bite. The same brownie bite with the recorded weight was used to perform the TD-GC–MS analysis to confirm the theoretical THC value according to the label.

To calculate and confirm the amount of THC, EGA was first performed on a THC standard (Absolute Standard). The pyrolyzer furnace was programmed from 100 °C to 800 °C (20 °C/min), and the EGA thermogram as shown in Figure 1 was obtained.

From the EGA thermogram, the optimal thermal desorption zone of THC was identified as 100 °C to 300 °C. In fact, using the MS interpretation library, the peak with the apex of 185 °C (100 °C to 300 °C temperature zone) was identified as THC. TD-GC–MS analysis was then performed on the brownie bite in triplicate as shown in Figure 2. To perform TD-GC–MS analysis, the pyrolyzer furnace was programmed from 100 °C to 300 °C (100 °C/min) after the EGA tube was replaced by a separation column (easily facilitated by using the vent-free GC–MS adaptor). The amount of sample used to obtain the TD chromatograms was 0.25–0.26 mg.

The peak shown in Figure 2 (noted with a *) between the retention time of 12 to 14 min was identified as THC using the MS interpretation library. The percent relative standard deviation (RSD%) of 4.6% was calculated based on the area counts of the THC peak. The most intense peak around 6 min was identified as 5-hydroxymethylfurfural (dehydration of sugar).

To confirm the THC concentration in the brownie bite, a standard addition calibration curve was created. The standard solution was a mixture of cannabidiol (CBD), THC, and cannabiol (CBN). Figure 3 shows the calibration curve and
calculated amount of THC in the brownie bite.

Using the calibration curve, the THC concentration is calculated as 0.996 mg/g while the product label indicates 1 mg/g of THC.

**Sample 2**

A commercial cannabis-infused dark chocolate bar with the total of 100 mg THC and net weight of 50 g (1.7 oz) was used for sample 2. According to the product label, there are 20 pieces of chocolates and each piece of chocolate contains 5 mg of THC, so there is 100 mg/50 g = 2 mg/g of THC present in each piece. To demonstrate the accuracy and precision of the methodology, the analysis was performed in triplicate. The weights of each piece were recorded using an analytical balance as 0.099 mg, 0.097 mg, and 0.105 mg.

The same methodology was used for analyzing sample 2. First, the EGA was performed as the fast rapid screening technique. Then the optimal thermal desorption zone of THC was identified. The pyrolyzer furnace was programmed from 100 °C to 300 °C (100 °C/min) to obtain the thermal desorption chromatograms for all three pieces of the chocolate. Figure 4 shows the TD chromatograms in triplicate.

The peak shown in Figure 4 (noted with a *) between the retention time of 12 to 14 min was identified as THC using the MS interpretation library. The RSD% of 4.6% was calculated based on the area counts of the THC peak.

To confirm the THC concentration in the chocolate pieces, a standard addition calibration curve was created. The standard solution was a mixture of CBD, THC, and CBN. Figure 5 shows the calibration curve and calculated amount of THC in the pieces.

Based on the calibration curve, the THC concentration is calculated as 2.1 mg/g while the product label indicates 2 mg/g of THC.

**Sample 3**

A commercial cannabis-infused gummy package with the net weight of 45 g (1.59 oz) was used for sample 3. The package contains 10 gummies as 2.1 mg/g while the product label indicates 2 mg/g of THC.

TO CONFIRM THE THC CONCENTRATION IN THE CHOCOLATE PIECES, A STANDARD ADDITION CALIBRATION CURVE WAS CREATED. THE STANDARD SOLUTION WAS A MIXTURE OF CBD, THC, AND CBN. FIGURE 5 SHOWS THE CALIBRATION CURVE AND CALCULATED AMOUNT OF THC IN THE PIECES.

Based on the calibration curve, the THC concentration is calculated as 2.1 mg/g while the product label indicates 2 mg/g of THC.

**Sample 3**

A commercial cannabis-infused gummy package with the net weight of 45 g (1.59 oz) was used for sample 3. The package contains 10 gummies as 2.1 mg/g while the product label indicates 2 mg/g of THC.

**CAN-TERPE-KIT**: Terpenes at 100 μg/mL in Methanol (42 components) or **CAN-TERPE-KIT-H**: Terpenes at 1,000 μg/mL in Methanol (42 components)

**CAN-CAN-KIT**: Canadian Cannabis Pesticide Kit at 100 μg/mL in LC/MS Acetonitrile.
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According to the product label, each gummy has 10 mg CBD and 5 mg THC. The label also indicates that tetrahydrocannabinolic acid (THCA), cannabidiolic acid (CBDA), and CBN (other required cannabinoids) are 0.0 mg/package.

To perform the TD-GC–MS analysis, one of the gummies was weighed on an analytical balance. The weight was recorded as 0.109 mg. The sample was then placed in MeOH and sonicated for 1 h. Next, 1 μL of MeOH solution was spiked into the sample cup.

It is important to mention that the gummy sample could be analyzed as is without dissolving in any solvent. The experiment on sample 3 was performed based on external standard calibration to demonstrate the reliability and flexibility of this technique.

Figure 6 shows the TD-GC–MS chromatograms of the gummy samples in triplicate. The EGA thermogram of the gummy confirmed the thermal zone of 100 °C to 300 °C for both THC and CBD. Note the detection peaks of both CBD and THC in the chromatograms. The RSD% of 2.5% for CBD and 2.3% for THC are calculated based on the area counts of the THC peak.

To confirm the THC and CBD concentrations in the gummy sample, an external standard calibration curve was created. Figure 7 shows the calibration curves and the calculated concentrations, which are in excellent agreement with the package label. CBD concentration is calculated as 8.5 mg/gummy compared to the label value of 10 mg/gummy. The calculated THC is 6.1 mg/gummy while the label indicates the value of 5 mg/gummy.

Table 1: Summary of the theoretical and calculated cannabinoids in the edibles

<table>
<thead>
<tr>
<th>Target compound</th>
<th>Brownie</th>
<th>Chocolate Bar</th>
<th>Gummy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Product label</td>
<td>THC</td>
<td>THC</td>
<td>CBD</td>
</tr>
<tr>
<td>Calculated result</td>
<td>1 mg/g</td>
<td>2 mg/g</td>
<td>10 mg</td>
</tr>
<tr>
<td>Repeatability (N = 3) RSD (%)</td>
<td>0.996 mg/g</td>
<td>2.1 mg/g</td>
<td>8.5 mg</td>
</tr>
<tr>
<td>Calibration curve (R²)</td>
<td>0.998</td>
<td>0.994</td>
<td>0.999</td>
</tr>
</tbody>
</table>

1-yl]-5-pentybenzene-1,3-diol). According to the product label, each gummy has 10 mg CBD and 5 mg THC. The label also indicates that tetrahydrocannabinolic acid (THCA), cannabidiolic acid (CBDA), and CBN (other required cannabinoids) are 0.0 mg/package.
curves were used for the brownie bite and chocolate piece while an external standard calibration curve was created to calculate THC and CBD in the gummy sample.

In conclusion, thermal desorption analysis eliminates conventional sample preparation regimes; the sample is heated to the point that the cannabinoids desorb from the edible matrix (thermal extraction). It is fast, uses minimal or no solvent, and eliminates the need for expensive glassware. TD-GC–MS is a “volatiles only” analysis; high boiling sample constituents remain in the sample cup which eliminates system contamination, increases system stability, and reduces run-to-run analysis time.

A multimode pyrolyzer provides users with a clear picture of the sample’s composition by identifying the thermal zones and the compounds in each zone. Using the EGA thermogram, one can simply determine the suitable temperature program and program the pyrolyzer’s furnace appropriately.

Using pyrolysis GC–MS, solid samples can be analyzed. There is no solvent required when using pyrolysis-thermal desorption GC–MS as opposed to traditional GC–MS techniques. In other words, the solid and liquid samples can be injected (using an inert sample cup) into the pyrolyzer without any solvent and sample pretreatment. This advantage, as well as rapid screening capability, is one of the primary reasons many laboratories integrate multimode pyrolyzers into both their day-to-day quality control and analytical research protocols.

Pyrolysis GC–MS techniques also enable material characterization of virtually any organic material by providing detailed information about the composition of the samples. This technique is used for identification of complex polymers, copolymers, volatiles, and additives as well as failure, contamination, deformation, and degradation analyses. As opposed to GC–MS alone, pyrolysis GC–MS allows multiple and customized analysis on the same sample while the MS libraries offer a broad range of references: from low boiling point compounds to pyrolyzates and heavier polymeric fragmentations. In terms of the ability to run samples at higher temperature, pyrolysis GC–MS can provide the user with an expanded temperature range from ambient +10 to 1050 °C (±0.1 °C precision) as well as a high interface temperature (maximum 450 °C).

**Acknowledgement**
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**References**

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Major Engine of Growth in the Cannabis Industry: State of the Art Formulation Technology for Product Innovation and Expanded Applications

Formulation technologies provide a powerful tool in product innovation and new applications especially for purified and highly concentrated extracts including cannabidiol (CBD) and tetrahydrocannabinol (THC). Fortunately, the cannabis industry can choose from formulation technologies and ingredients that have a record of use in pharmaceuticals, food and beverages, dietary supplements, personal care, and veterinary and pet food products. This article aims to assist in making choices that support safety, efficacy, stability, and consumer acceptance.

Andreas M. Papas

New, innovative products and applications have been a major driver of growth of the cannabis industry. The range and number of new products is breathtaking and spans pharmaceuticals, food and dietary supplements, topical and personal care, and veterinary and pet food. It includes product formats ranging from prescription pharmaceutical formulations to capsules, chewable tablets, nutrition bars, gummies, drops, bottled water, beverages and sports drinks, creams, lotions, sprays, inhalation and vapor products, and pet snacks and treats.

In a significant milestone, in June 2018 the U.S. Food and Drug Administration (FDA) approved the drug Epidiolex (GW Pharmaceuticals), which is an oral solution of cannabidiol (CBD) for the treatment of seizures associated with two rare and severe forms of epilepsy. Epidiolex is the first FDA-approved drug that contains a purified drug substance derived from cannabis—in a strong pipeline of development and clinical testing.

Purified and concentrated cannabis extracts are powerful tools in product innovation. For medical applications, they allow the identification of the active ingredients, clinical testing for FDA approval and claims, and products that have defined potency and can be tested with established scientific methods for meeting specifications with
consistency and stability. The same reasons increasingly apply to dietary supplements, beverages and sports drinks, and other applications. The use of purified and concentrated extracts also allows for the use of smaller amounts, which has significant benefits for taste, odor, color, or capsule and tablet size for oral supplements. The use of purified and concentrate extracts also facilitates accurate testing using smaller sample size.

Why Formulation: The Top Reasons
Formulation is key to new innovative products and applications for the following reasons:
- Solubility in water
- Increased absorption and bioavailability
- Improved stability
- Taste, odor and other characteristics
- Regulatory
Let’s take a closer look at each reason below.

Solubility in Water
Upon extraction and purification, important cannabinoids such as CBD and tetrahydrocannabinol (THC) come out as oils that are practically insoluble in water. For many products and applications such as beverages, medical formulations, sprays, topical formulations, and others, solubility in water is a must.

Increased Absorption and Bioavailability
Absorption of oils requires optimal function of the liver, pancreas, and the complete digestive tract. Absorption is also affected by the diet, age, and health conditions of the user. Clinically proven formulation technologies increase absorption and bioavailability especially in the large population segments with suboptimal absorption (1,2).

Improved Stability
The stability and shelf-life of cannabinoids is affected by exposure to sunlight, air, heat, and other factors. When cannabinoids are added to water, foods, creams, and other products, their stability can be affected by exposure to oxidizing agents, acidic or alkali conditions, and reactive compounds. Customized formulations provide powerful tools for optimizing stability.

Taste, Odor, and Other Characteristics
Taste is of paramount importance
in food, beverages, and other oral products. Odor is also important. Terpenes, which are abundant in cannabis plants, can have strong aromas that may be carried over into extracts, concentrates, and even purified cannabinoids. Formulation provides the opportunity for addressing the taste, odor, texture, and other characteristics for the specific products and applications.

**Regulatory**

For medical applications, formulation is required for accurate dosing of the desired product. With the expanding approval of cannabis products in the United States, Canada, and other countries, there is increasing focus on quality, testing and product specifications, potency, stability, and other product characteristics.

**Choosing Formulation Agents: Top Criteria**

The top criteria to consider when choosing the right formulation agents include safety, efficacy, positive or neutral effect on important properties, and support of label and customer preferences. Let’s explore each of those criteria in more detail.

**Safety**

The FDA’s GRAS (generally recognized as safe) designation is a top selection criterion. A long record of safe use on a daily basis, with significant safety margins across wide dose ranges supported by clinical evidence, provides additional confidence.

**Efficacy**

This criterion requires major homework, especially for the critical parameters of water solubility, stability, and increased absorption and bioavailability. While efficacy claims are abundant, few are supported by evidence. The gold standard is research and clinical evidence from leading universities and researchers, preferably supported by the National Institutes of Health (NIH) or similar quality funding organizations, and the results published in refereed scientific and medical journals.

**Positive or Neutral Effect on Important Properties**

This includes taste, odor, and color. For oral products, especially foods and beverages, taste and odor are of paramount importance.

**Support of Label and Customer Preferences**

Although there is no official definition of clean label, consumers increasingly show preference for minimally processed products with safe ingredients, preferably derived from natural sources. Additional consumer preferences include no sugar or salt added, free of gluten, dairy, and animal products, nuts and other sources of allergens, and nongenetically modified organisms (non-GMO).

**Key Commercial Formulation Applications**

The major cannabinoids, CBD and THC, are key components of innovative products. Increasingly, these cannabinoids are used in concentrated or purified form, especially for medical, dietary supplement and food and beverage applications, sprays, and topical products. Key objectives of formulation were discussed above and include:

1. Solubility in water especially for the emerging market segments of infused water, beverages, sports drinks, and other water-based products.
2. Increased absorption and bioavailability, which is key.
glycols, and glycerin support the formulation of liquids.

The increasing consumer awareness and strong preference for simple formulations using ingredients with proven records of safety and efficacy, and the major role of taste and odor, especially for oral products, have limited the practical choices dramatically. For example, polylsorbates (such as Tween-80) which are common, strong solubilizers in the pharmaceutical, food and beverage, and cosmetic industries, can impact the taste. More importantly, an increasing consumer segment prefers products that do not contain polysorbates (4).

Vitamin E TPGS, a derivative of naturally sourced alpha-tocopherol is becoming the formulation ingredient of choice for cannabis products, especially purified or concentrated CBD and THC and other extracts. The reasons include:

- A proven record of safety (1,5) based on decades of commercial use in pharmaceuticals and dietary supplements, including vitamin formulations for children.
- Efficacy based on conclusive clinical evidence (6), not only as a solubilizer for water-soluble formulations, but also in its ability to form micelle-like particles that enhance absorption and bioavailability. Its efficacy is illustrated by its use in clinically-tested (7), FDA-approved products formulated for enhanced absorption.
- Neutral taste and odor and light yellow color are particularly important for oral and personal care product formulations.
- Support of product stability. Vitamin E TPGS contains some free tocopherols that have strong antioxidant activity and support product stability especially for oil-based products. In addition to reducing potency, products of oxidation affect taste, odor, and can be harmful.
- Support of label claim of free of allergens, sugar, salt, gluten, dairy, and animal products.

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4) http://www.ewg.org/skindeep/ ingredient.php?ingred06=705142& refurl=%2Fproduct.php%3Fprod_id%3D62813%26f%3FWt6DvZnRfyQ.

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It seems most analytical scientists have an “ah ha!” moment when they realize the incredible opportunities in cannabis science. When did you start to see these opportunities, and how did you first get involved in cannabis science?

Professor Jack Henion: I actually had two “ah ha” moments. The first was as an analytical chemist I was intrigued to see how analytical methodologies and technologies used for cannabis were not as technologically current as those in the good laboratory practice-regulated pharmaceutical bioanalytical world with which I was familiar. This coupled with the chemical complexity and diversity of the many cannabis cultivars and the importance of accurate and reliable analyses suggested that perhaps I could contribute to improving the analytical sciences used in this growing industry. However, the real excitement, or second “ah ha” moment for me, occurred when I attended the first Cannabis Science Conference in 2016 where I witnessed compelling testimonials of the very positive medicinal marijuana benefits afforded to certain disease sufferers. A common successful business model is to “find a need and fill it.” I was convinced this was an exciting new field where I could perhaps contribute to “filling a need” by providing positive contributions to supporting the scientific integrity of this new industry through high-quality analytical techniques and technology. I remain excited about working within and contributing to this fast-paced, evolving industry.

Every scientist that I meet with struggles in some way with analyzing cannabis. What hurdles did you first experience in working with cannabis and how did you overcome them?

Henion: Our experience with marijuana plant materials to-date is somewhat limited because we do not yet have a Schedule I controlled substances license from New York State. We have applied for this license, but do not yet have it. As a result, I have focused on the analysis of hemp plant materials and products which of course must contain less than 0.3% tetrahydrocannabinol (THC). I would not say that the analysis of these plant materials is particularly difficult. In contrast to my experience with parts-per-billion (ppb) and sub-ppb bioanalysis of drugs and their metabolites in biological fluids, quantifying the important cannabinoids in hemp and marijuana is relatively easy because the levels are much higher (percent levels) and the matrix is much less complex than blood or urine. In contrast, the accurate and precise quantitative determination of as many as 100 pesticides at low ppb levels in marijuana plant materials is of course more challenging. Plant sample preparation when handling large sample numbers needs some advancement, but the technologies and methods available when properly applied are not that difficult. Of course, there are additional quantitative measurements needed such as for terpenes, flavonoids, mycotoxins, and heavy metals, which in some cases require different sample preparation techniques and instrumentation. Thus, a “total analysis” of a cannabis plant sample is not just one sample preparation technique followed by an injection into one analytical system. It is a series of related, but different procedures.

You have started lecturing on cannabis and hemp at Cornell University. What were some of your takeaway messages and how were those lectures received?

Henion: I recently gave a lecture to the Cornell BioEngineering MS graduate students where I focused on the importance and benefits of collaboration and innovation in science and business development. I described the rapidly developing cannabis industry as a new frontier with many technology development opportunities for these students in their bioengineering careers. I received many inbound inquiries after this lecture, which suggests I struck a chord of interest and excitement with these students by sharing with them some industry intellect regarding opportunities in the cannabis and hemp industries.
Additionally, I recently presented two lectures at the annual Cornell Veterinary Conference at the Cornell University College of Veterinary Medicine, Ithaca, NY (1). There is growing interest and acceptance, especially among companion animal veterinarians for the use of cannabidiol (CBD) oils and related products to manage pain and other ailments for their “patients.” These lectures included a “tutorial” on liquid chromatography–mass spectrometry (LC–MS) and related techniques showing methods used for the analysis of hemp and marijuana plant materials as well as a second lecture on the comparison of analytical results from commercial veterinary oils, tinctures, and so forth versus what is listed on the labels. My message was caveat emptor since often the product label does not accurately reflect what is in the bottle. The veterinarians in the audience appeared to be very interested in these topics.

As a pioneer and leader in mass spectrometry, can you please comment on its role in cannabis science?

Henion: I believe mass spectrometry is an essential component for the credible growth and acceptance of the cannabis industry. I, of course, can be accused of being “overly prejudiced” in my interest and support of MS as a detector and, in particular, LC–MS techniques. In my early days of racehorse drug testing where our analytical results would have to stand up to legal scrutiny in court, my students used to call the mass spectrometer the “truth machine.” Currently, there is no other detector for the determination of organic compounds that provides the sensitivity, selectivity, and speed for the money than MS. When MS is coupled to gas chromatography (GC) and for even wider applications, LC (or best with ultrahigh-pressure liquid chromatography [UHPLC]), one has unequaled analytical capabilities. I worry a bit about the current unchallenged interest in LC–photodiode-array detector (PAD) for cannabis potency determinations because we know there are chemical entities in the sample matrix that can coelute or interfere with photodiode-array detection. If LC–MS techniques could be competitively priced and coupled with ease-of-use, I would prefer to use those techniques for potency determinations rather than LC–PAD. But that should be left up to the individual laboratory and its leadership.

Please comment on what is next for you in cannabis science. What business and research projects are you working on that you can share with us?

Henion: As you may perceive from my comments above, I am very excited to participate in and hopefully contribute to maintaining a high degree of scientific excellence used in the analysis of any cannabis-related samples. This pertains to plant materials for potency and pesticides as well as the rapidly increasing diverse array of commercial products. I have heard reference to the “wild, wild west” nature of some current procedures and policies in the medical marijuana world. The days of “snake oil” sales pitches and unsubstantiated product promises should not return because it will diminish the credibility and potential of this exciting field. I have proposed that Advison, Inc., establish a “Cannabis CRO” and plans for that are underway. It is my goal to establish and maintain a state-of-the-art laboratory that maintains the highest level of scientific analytical services. In the meantime, we are carrying out applied research in sample preparation and LC–MS analysis techniques for hemp-based plants and products to improve upon analytical procedures currently used. To have a ready source of fresh hemp plant materials, this past summer I qualified as an approved “Cornell Affiliate” hemp grower under a permit issued by the New York State Agriculture and Markets and collaborated with Cornell University to grow a small hemp plot on my personal farm near Ithaca, NY. So, now I know a little bit about growing and analyzing hemp.

Are there any additional comments you would like to make regarding the future of cannabis science?

Henion: I believe medicinal marijuana and its promising industry will be part of the future. However, we have a very steep learning curve ahead of us. Academic research is thwarted because of misguided opinions and funding is limited for similar reasons. The good news is, I believe, this will change with time and that in fact there are many exciting opportunities ahead. My plea is for the maintenance of scientific and business integrity such that the benefits and opportunities remain available for all. As Nike says, let’s “just do it.”

Reference

About the Interviewee
Dr. Jack Henion is an internationally recognized leader in the field of MS and LC–MS. He is credited with 13 patents and has published more than 200 peer-reviewed papers in scientific journals. In addition to vast industrial experience, Dr. Henion managed a major research laboratory at Cornell University, where he served as a professor of toxicology for more than 24 years and is now Emeritus Professor of Analytical Toxicology.

About the Columnist
Joshua Crossney is the columnist and editor of “Cannabis Crossroads” and a contributing editor to Cannabis Science and Technology magazine. Crossney is also the president and CEO of C3C Events. Direct correspondence to: josh@jcanna.com
The Advantages of an Industry-Specific LIMS for Cannabis Labs

Marty Pittman, Senior Product Manager, LabVantage Solutions Inc.

LabVantage Cannabis is a purpose-built informatics platform, offering cannabis testing labs approximately 80% of commonly required industry functionality.

People in industries that have long been using laboratory information management systems (LIMS) remember the days (or months or years) of customizing a base software platform into a workable solution specifically for their organization. The emerging cannabis industry is now benefiting from these years of customization, as LIMS vendors have begun building more of the common, necessary functionality into the base product.

Industry accelerators take this approach a step further; the LIMS platform is purpose-built for cannabis testing labs, offering right out-of-the-box approximately 80% of commonly required functionality and, for the remaining 20%, it’s easily configured without the need for coding. The biggest advantage of purpose-built software is the speed of deployment, and the subsequent reduced cost and risk. Industry accelerators can cut implementation time by as much as 75%, and savings by about the same.

Without the need to spend time analyzing workflows and processes to determine system requirements, the accelerated LIMS gets right to helping reinforce consistent, industry-standard workflows that lead to greater efficiency and productivity. LIMS prepackaged with electronic lab notebook functionality and industry-specific modules create a streamlined system that maximizes return on investment while lowering risk because custom code is not needed. With an interface that is responsive to the specific industry, the intuitive purpose-built LIMS reduces the time and investment in user training.

What to Look for in an Industry-Specific LIMS Grower Portal

Because cannabis testing labs serve a wide range of growers, a portal into the system is a desirable feature. It provides growers with a frictionless mechanism for preparing and submitting requests for the lab.

Growers must be able to easily enter details about the sample; specify which tests are needed and when; provide details such as METRC ID or the mass of each batch; and track all of their harvest lot requests at various stages. The system must deliver a certificate of analysis that is modifiable for different lab results.

Tracking Capabilities

Both growers and lab administrators as well as lab managers want visibility to lab processing. Lab leaders must be able to drill down into the details of particular requests, and automatically send any questions back to growers. Password-protected electronic signatures are required throughout the process to maintain a digitized chain of custody.

METRC Integration

As samples are received and processed, the LIMS should interface with METRC to signal that the sample is undergoing testing. Likewise, the system should share results and auto-populate invoices. Such integration contributes to a more efficient workflow between the grower, the lab, and regulators.

Industry-Standard Testing

A cannabis LIMS must support industry-standard testing, such as American Herbal Pharmacopoeia-recognized analytical test methods.

A purpose-built cannabis LIMS works in lockstep with lab teams, from initial request through final invoicing, in a way and at a deployment speed that traditional LIMS cannot. The industry-accelerated LIMS enables a highly efficient and scalable workflow supported by industry-standard quality control.

For a white paper and information on LabVantage Cannabis, a cannabis-specific LIMS platform, visit labvantage.com/cannabis.
2018 Supplier Profiles Issue
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Chem Service, Inc., produces high purity chemicals for use as reference materials and for other laboratory purposes. More than 95% of their standards grade materials have a certified purity of 98.0% or greater. Standards-grade chemicals are clearly labeled with an expiration date that is based on years of experience in handling and testing. Products are packaged in small quantities to minimize storage, waste, and disposal requirements. Organic and inorganic chemicals, solutions, and mixtures are available to meet a wide range of specialized laboratory needs.

MAJOR PRODUCTS
- Pesticide residue standards
- Cannabis pesticide standards
- Residue solvent standards
- Flame retardants/PBDE
- Metabolite standards
- EPA standards
- USP standards
- ISO standards
- DIN standards
- Explosive residues
- PCB congeners
- Petroleum hydrocarbons
- Fatty acid methyl esters
- High purity inorganics
- Surfactants
- Anti-oxidants
- Ultraviolet inhibitors
- Plasticizers
- Biological stains
- Phthalate standards
- Polynuclear aromatics
- Vitamins
- Carbohydrates standards
- Forensic standards
- Personal care product standards

STATES SERVED
US and international

Chem Service, Inc.
600 Tower Lane
West Chester, PA 19380
USA

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Toll free: (800) 452-9994
(610) 692-3026
Fax: (610) 692-8729

EMAIL
info@chemservice.com

www.chemservice.com
Frontier Laboratories

Edibles quick and easy by EGA/PY 3030D Multi-Mode Pyrolyzer

COMPANY DESCRIPTION
Frontier Laboratories, Ltd., was founded in 1991 by Dr. Chu Watanabe. His experience working at Hewlett-Packard’s Analytical Division and Dow Chemical made him uniquely qualified to design and market analytical instruments for materials characterization. Dr. Watanabe, with the support of polymer scientists at Nagoya University in Japan, developed a pyrolyzer based on a vertical microfurnace design. Today the patented fourth generation vertical microfurnace serves as the cornerstone for the 3000 Series of products developed and marketed worldwide by Frontier Laboratories.

CHIEF SERVICES SUPPORTED
Research, development, and manufacture of analytical equipment.

MAJOR PRODUCTS
Frontier Laboratories, Ltd., designs and manufactures analytical instruments for materials characterization. The main products, supported by a number of accessories and software, include the EGA/PY-3030D Multi-functional Pyrolysis System, the PY-3030S Single-Shot Pyrolyzer, the Rx-3050 series of Rapid Screening Reactors for catalyst screening, and a line of Ultra ALLOY® stainless steel capillary columns. Frontier Labs’ products are compatible with most gas chromatographs and mass spectrometers from major manufacturers.

STATES SERVED
Nationwide for the U.S. and Canada, as well as Asia/Oceania, Europe, Middle East/Africa, and Russia.
Restek Corporation

COMPANY DESCRIPTION
For more than 30 years, Restek has been a leader in developing technologies and manufacturing products for gas and liquid chromatography (GC and LC), including columns, reference standards, sample preparation materials, accessories, and more. We have decades of hands-on practical experience in chemistry, chromatography, and engineering, and our reputation for going the extra mile with our Plus 1 customer service and top-performing products is well known throughout the chromatography community. Restek has been providing the cannabis market with testing consumables and services since 2010 for both product safety and product quality. This includes supporting testing for potency, pesticides, residual solvents, mycotoxins, and terpenes.

CHIEF SERVICES SUPPORTED
Whether you are part of a well-established quality assurance lab or starting a new lab, Restek has the products and expertise you need for successful cannabis analysis. Being an employee-owned and independent chromatography company, every employee at Restek has a vested interest in your success. We design the best solutions for your lab, regardless of the instrumentation and techniques used.
- LC
- GC
- Sample preparation
- Reference standards

MAJOR PRODUCTS
We offer a range of chromatography consumables for potency, pesticides, residual solvents, mycotoxins, and terpenes. Our chemists develop LC and GC columns, reference standards, and sample preparation products that streamline cannabis testing. Restek’s Rxi columns deliver more accurate, reliable results than any other fused silica column on the market. Our Raptor LC columns are ideal for cannabis testing because they quickly separate your target compounds, providing higher sample throughput. Restek is also continually expanding our product line in order to meet the evolving needs of the cannabis industry. Restek’s certified reference materials are manufactured and quality control tested under ISO 17034 and ISO/EIC 17025 accreditations.

STATES SERVED
Nationwide and North America.
Shimadzu Scientific Instruments

COMPANY DESCRIPTION
Shimadzu is one of the world’s leading providers of analytical instruments for applications in a broad range of industries. In the United States, Shimadzu has been at the forefront of working with cannabis testing laboratories and has a comprehensive understanding of the requirements of this evolving industry. From seed to sale, from accurate potency testing and terpene profiling to contaminant testing for pesticides, residual solvents, heavy metals, and mycotoxins/aflatoxins, Shimadzu offers scalable solutions to meet your testing needs for today and tomorrow.

CHIEF SERVICES SUPPORTED
As medicinal and recreational cannabis markets continue to grow, analytical testing will ensure that consumers are receiving accurately labeled products that are free from contamination. Shimadzu is ready to assist you as you grow your laboratory. We offer a full suite of testing instruments, research platforms, scientifically validated methods, and a variety of leasing programs to meet evolving requirements. In addition, our expert team of scientists is readily available to help your cannabis testing laboratory succeed, assisting with method development, instrument training, and maintenance to ensure your systems operate at an exceptional level.

MAJOR PRODUCTS
Shimadzu offers a full suite of analytical instrumentation to help grow your lab. These instruments include:
• HPLC
• GC
• GC–MS/MS
• LC–MS/MS
• AA
• ICP-MS
• Moisture balances
• Online SFE-SFC-LC–MS/MS
• MALDI-TOF MS
• Consumables

STATES SERVED
Nationwide
Pixis Labs

COMPANY DESCRIPTION
Pixis Labs sets the standard for quality analytics in the CBD and hemp markets nationwide and raises the bar for Oregon cannabis laboratories. In partnership with Columbia Food Labs, and as a member of Tentamus Group, Pixis Labs boasts top-of-the-line instrumentation and nearly 40 years of analytical expertise. With low detection limits and robust methodologies, Pixis Labs is ahead of the curve for changing requirements and emerging markets, offering services beyond compliance. We pride ourselves on building a community of sustainable business partnerships through detailed analyses and accurate reporting. Contact us for a custom service and price quote.

CHIEF SERVICES SUPPORTED
Pixis Labs offers you reliable data and turn-around times with transparent, professional communication and competitive pricing.
- Potency HPLC (ISO 17025)
- Pesticides by LC–MS (ISO 17025)
- Heavy metals by MS/MS (ISO 17025)
- Residual Solvents by GC–MS (ISO 17025)
- Organic-equivalent pesticide screen (ISO 17025), developed in-house, by LC–MS
- Mycotoxins by LC–MS/MS (ISO 17025)
- Moisture, water activity
- Terpenes by GC–MS
- Pathogens
- Yeast and mold
- Nutritional lab: FDA Labels, allergens, vitamins, supplements, shelf life, and more

MAJOR PRODUCTS
Pixis Labs offers accurate and clear reporting, catered to your market and industry.
Consultation services are available upon request.

STATES SERVED
Pixis Labs’ services are available nationally for hemp and CBD analyses, as well as nutritional, environmental, and microbiological testing. We are proud to serve the Oregon cannabis market for compliance and R&D testing.

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KEY PERSONNEL
Derrick Tanner, General Manager
Jen Stiles, Operations Manager
Kelly O’Connor, Sales Rep

www.columbiafoodlab.com

Pixis Labs
A Tentamus Company
SPEX CertiPrep

COMPANY DESCRIPTION
SPEX CertiPrep has been servicing the scientific community since 1954. We are a leading manufacturer of certified reference materials (CRMs) and calibration standards for analytical spectroscopy and chromatography. We offer a full range of inorganic and organic CRMs. We are certified by DQS to ISO 9001:2015 and are proud to be accredited by A2LA to ISO/IEC 17025:2005 and ISO 17034:2016. The scope of our accreditation is the most comprehensive in the industry and encompasses all our manufactured products.

MAJOR PRODUCTS
We offer analytical standards for medicinal and recreational cannabis testing. SPEX CertiPrep offers ISO 17034 CRMs for all of the common contaminants such as pesticide residues, residual solvents, and heavy metals as well as qualitative analysis CRMs such as terpenes and cannabinoids. As the industry demands change and regulations are put into place, we continually update our product offerings.

STATES SERVED
Nationwide for the US and Canada.
VividGro

COMPANY DESCRIPTION
VividGro is a pioneer in the AgTech space. Tailored to support the automation and efficiency needs of the indoor agriculture and horticultural markets, VividGro implements solutions that help growers maximize yields and reduce costs. The state-of-the-art, customized lighting product line delivers optimized PAR to maximize plant growth and PAR efficacy. Our control automation systems and data collection strategies are able to measure and modify growing to help growers learn how to use their resources more efficiently. It is not the lights you use; it is how you use them. Learn more at VividGro.com.

CHIEF SERVICES SUPPORTED
• Custom LED grow lights, automation, and controls

MAJOR PRODUCTS
• GroBar X (single channel)
• GroMax (3 channel)
• FlowerMax (6 channel)
• FlowerMax Pro (8 channel)
• GroNet (automation and control system)

STATES SERVED
Nationwide

VividGro
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LOCATIONS OF OTHER OFFICES AND FACILITIES
Cocoa Beach, FL

KEY PERSONNEL
Randy Shipley, VP of Sales
David Friedman, CEO
Dawn Pogue, Customer Experience Manager

vividgro.com
FRITSCH Milling & Sizing, Inc.

COMPANY DESCRIPTION
FRITSCH is an internationally respected German manufacturer of laboratory and production-scale instruments used for milling, particle size analysis, and sample handling. Serving a broad range of industries, including food, pharmaceuticals, agriculture, and nano-technology, FRITSCH has worked closely with the medical cannabis industry to optimize a range of solutions for use in production, quality control, and R&D.

PRIMARY APPLICATIONS SUPPORTED
- Production milling—optimize particle size to maximize extraction yield, homogenize solid extracts and ingredients, create Premium prerolls.
- Quality control—homogenize edibles and plant samples to allow representative subsampling
- Particle size analysis of milled materials

MAJOR PRODUCTS
- PULVERISSETTE 19—Precision cutting mill system
- PULVERISSETTE 0—Vibratory micro mill with cryoBox
- ANALYSETTE 22 NanoTec—Laser particle size analyzer

REGIONS SERVED
Global

FRITSCH Milling & Sizing, Inc.

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LOCATIONS OF OTHER OFFICES AND FACILITIES
Global Headquarters: Idar-Oberstein, Germany
Company Offices: USA, France, Russia, Singapore, China
Local agency representatives in all world regions

www.fritsch-us.com
www.cannabis-milling.com

www.CannabisScienceTech.com | CANNABIS SCIENCE AND TECHNOLOGY
LGC Standards

COMPANY DESCRIPTION
LGC Standards is a global manufacturer and distributor of high quality reference materials with a portfolio of more than 100,000 certified reference materials, standards, and proficiency testing schemes for the pharmaceutical (Mikromol™), environmental (Dr. Ehrenstorfer™), cannabis, food and beverage, forensic, clinical, industrial, and petrochemical markets. Our ISO accreditations include: ISO 9001, ISO 17034/ISO Guide 34, GMP/GLP, ISO 13485, ISO/IEC 17025, and ISO/IEC 17043.

As a designated National Measurement Institute (NMI) for chemical and bio-measurement, LGC works in partnership with governments, intermediaries, and private sector organizations to address the measurement needs of key industries including healthcare, food, environmental, security, and energy.

MAJOR PRODUCTS
Dr. Ehrenstorfer™ offers a broad range of state-by-state cannabis impurity standards as well as a full list of terpenes to meet your state’s requirements. We manufacture standards for the analysis of pesticide, solvent, heavy metal, mycotoxin, bacteria, and growth regulator residues. All of our Dr. Ehrenstorfer™ brand mixes are certified reference materials manufactured in accordance with ISO 17034 requirements. Custom mixes are our specialty.

STATES SERVED
Global

https://us.lgcstandards.com
PRO Scientific Inc.

COMPANY DESCRIPTION
PRO Scientific is a global leader in the manufacturing of homogenizers for both laboratory and industrial labs requiring sample preparation. PRO homogenizers are able to homogenize various forms of cannabis material fast and efficiently.

From micro sample volumes to larger multi-liter processing, there is a PRO Homogenizer to suit your needs. PRO Scientific even offers homogenizing solutions to address automated multi-sample homogenizing and OEM homogenizer needs too. All products manufactured by PRO Scientific are backed with over 25 years of technical experience, unmatched customer support, and made in the USA.

CHIEF SERVICES SUPPORTED
PRO Scientific is the ideal source for high quality homogenizing products for sample preparation within the cannabis industry, providing assistance for both small startup and established labs with their cannabis testing and quality control. PRO Scientific homogenizers are precision homogenizers to assist in determining and monitoring the cannabinoid potency and pesticide residue. PRO Scientific Homogenizers are also ideal for the extraction of cannabis for the development of cannabis infused products. Homogenization of cannabis samples allows for a stable emulsion to be created that is shelf stable.

MAJOR PRODUCTS
Our PRO Homogenizers and generator probes are precision designed to provide reliable, reproducible results that are required for this industry. A range of setups are available depending upon your budget and processing requirements. Whether high speed shearing to dispersing, homogenizing, emulsifying, mixing, or blending.

- Economical homogenizing package kits
- Handheld or stand mounted homogenizer models
- Digital and programmable benchtop homogenizer models
- Automated and semi-automated homogenizing systems
- Precision crafted generator probes

STATES SERVED
PRO Scientific supports labs in various industries that require sample preparation throughout the United States as well as internationally.

PRO Scientific Inc.
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KEY PERSONNEL
Holly Yacko-Archibald, Vice President
Brian Archibald, Business Development Manager

www.proscientific.com
Vitalis Extraction Technology

COMPANY DESCRIPTION
Vitalis Extraction Technology Inc.® (Vitalis) is a privately-owned, Kelowna-based engineering and manufacturing company, producing the highest-flowing industrial supercritical CO₂ extraction systems for the cannabis industry. Renowned for their reliability, scalability, and continuous operation, Vitalis systems are designed and manufactured in accordance with ASME and CSA Standards for Boiler, Pressure Vessel and Pressure Piping Code. The vessels are stamped with a CRN and NB registration number confirming that the vessels meet code and have been examined by an Authorized Inspector. With systems on three continents, Vitalis has the most deployments of industrial CO₂ supercritical extractors into the cannabis industry.

MAJOR PRODUCTS
Vitalis Extraction Technology is the leading manufacturer of the most sophisticated industrial Supercritical CO₂ extraction systems in the world. Vitalis is Canada’s first and only American Society of Mechanical Engineers (ASME) certified extraction original equipment manufacturer (OEM) in the cannabis industry. Vitalis’ award-winning systems focus on the key needs of any large-scale extraction business: reliability, component certification, and continuous 24/7 operation. Vitalis systems were engineered to balance all three fundamentals, and the result is the highest output of the purest full-spectrum oil possible in the shortest amount of time in the CO₂ extraction industry.

Vitalis produces three lines of CO₂ extraction equipment: The F-Series, the Q-Series, and the R-Series. The R-Series’ R-400 allows for processing of up to 500 pounds of top-quality material per day. With a heavy-duty hydraulic pump, dual extraction lines, industrial grade CO₂ recovery system, TrueCyclonic separation technology, and modular assembly for future scalability, the Vitalis system is built for heavy-duty workloads, massive capacity, and continual growth. With a strict focus on quality as well as a commitment to constant research and development, Vitalis provides certified Supercritical CO₂ extraction equipment that is redefining the capabilities of traditional extraction methodologies.

STATES SERVED
Global—systems deployed across North America, South America, and Europe.
COMPLED Solutions GmbH

COMPANY DESCRIPTION
COMPLED was founded in 2010 and has become one of the pioneers in LED horticulture lighting. Since then COMPLEX has been in the forefront of LED horticulture lighting for science applications. We develop, manufacture, and test exclusively in Germany. We have always been focused on tunable light spectra because that’s the major advantage of LED technology. By dynamically tuning the spectrum it is possible to optimize yields as well as cannabinoid and terpene profiles. Furthermore, it is also crucial to align lighting recipes to all other relevant environmental parameters such as CO2, temperature, and so forth, and that’s actually also possible with our technology.

CHIEF SERVICES SUPPORTED
Besides horticulture lighting design we also offer support in the development of lighting and growing recipes, data science, and biological research related to lighting. Even customized lighting devices or spectra are available upon request. Please have a look at our references (www.compled.de/pages/references.html).

MAJOR PRODUCTS
Our main product line is called the SUNsim platform (www.compled.de/pages/products.html#research_jump). It was designed especially for researchers who are interested in the impact of the quantity and quality of light on the development of plants. We offer several solutions from 24 LED channels down to six LED channels.

In addition, we are proud to offer our first LED horticulture lighting platform especially designed for cannabis cultivation, the HashCropter platform (www.hashcropter.com). It’s the most flexible and effective LED lighting platform for cannabis cultivation.

Spectral tuning is always obligatory and both platforms are combinable.

STATES SERVED
Nationwide and worldwide.

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KEY PERSONNEL
Christoph Schubert, CEO

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LEAP PAL Parts + Consumables

COMPANY DESCRIPTION
LEAP PAL Parts + Consumables has been helping customers in the lab instrumentation and liquid handling business for more than 25 years. We represent the highest quality products that meet strict quality assurance (QA) standards and work with the best manufacturers to offer innovative products at the most competitive prices. We provide same day shipping of all in-stock products to avoid instrument downtime as well as reduce your on-hand inventory levels. Our mission statement is: Be Good. Just that simple. Be good to our customers, our suppliers, our community, and to each other. Let us show you what thousands of customers already know.

CHIEF SERVICES SUPPORTED
We offer a complete range of consumables for high pressure liquid chromatography, gas chromatography, headspace, and sample preparation. In addition to consumables, we offer instrument specific small parts. As an added service we also provide technical assistance by certified PAL, PAL-XT, PAL 3 service technicians if you need urgent assistance with your instruments.

MAJOR PRODUCTS
We provide syringes and syringe components, vials, caps and inserts including our own L-MARK brand, instrument parts and accessories, valve components including rotors, valves, tubing, fittings and sample loops as well as plates and cap mats. Our suppliers include CTC Analytics, VALCO Instruments, IDEX/Rheodyne, La-Pha-Pack and many others.

STATES SERVED
We serve all 50 states and Canada. Providing products internationally and domestically, to customers in the following industries:
- Biotechnology
- Pharmaceutical
- Clinical
- Food and beverage
- Environmental
- Petrochemical
- Cannabis analysis
- Forensics
- Toxicology

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KEY PERSONNEL
Lamar Jones, President
Kate Holub, VP of Sales
Angie Buchholz, Marketing Director

www.palparts.com
“There was an amazing energy level at this year’s show, a coming together of analytical, medical and cannabis experts that felt like an extended cannabis family!”

- Tracy Ryan (CannaKids and SavingSophie.org)

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EDIBLES
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- Virtually any sample can be chemically characterized.
- No solvent extraction and sample pretreatment needed.
- Only a small amount of sample is needed.
- Applicable to both production and research laboratories.
- The Pyrolyzer easily interfaces to a GC-FID and/or GC-MS system.

For all inquiries, product technical info, and our poster on analysis of cannabis sativa and edibles, contact rojin@frontier-lab.com (925) 813-0498

Frontier Labs North American Technology Center
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