Case study | 002364

Automated wet chemistry analysis

Frind Lab Services performs multianalyte soil analyses—and more—in minutes with the Gallery discrete analyzer

> "As a lab manager, I prefer Thermo Scientific instruments. The service is great, and the applications specialists are knowledgeable, patient, and help you understand how things work. Across the board everybody has been great. Even the sales reps are amazing, which is why I built a lab with Thermo Fisher Scientific equipment."
>
> —Tara Holitzki, Lab Manager, Frind Lab Services, Frind Estate Winery

Introduction

Soils are routinely analyzed to monitor fertility and guide the use of amendments essential to plant growth such as nitrogen, phosphorus, and potassium. Labs carrying out soil testing typically have numerous samples to analyze, making high-throughput automated approaches indispensable. Though most labs use a dedicated instrument for each soil test, instruments that offer the flexibility to perform many different tests are preferable to enable workload balancing, adjust to evolving priorities, and increase return on investment.

Frind Lab Services of Frind Estate Winery in West Kelowna, British Columbia (BC) is a BC Vintner's Quality Alliance (VQA) certified, fee-for-services lab that offers an extensive array of testing services, including soil testing for winemakers, farmers, orchardists, and researchers. The lab relies on state-of-the-art instrumentation to provide accurate and consistent results with 30-minute sample turnaround for most analyses. Notably, Frind Lab Services is at the forefront in applying the Thermo Scientific™ Gallery™ Discrete Analyzer to automated, high-throughput determination of sulfate, nitrate, ammonium, chloride, and phosphorus in soil samples.

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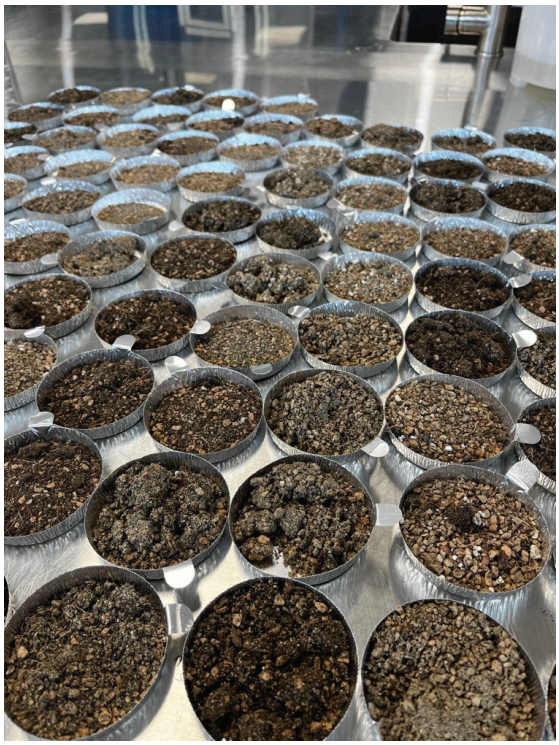
The Gallery discrete analyzers are integrated and highly automated to provide a faster and safer alternative to traditional wet chemistry methods. Designed to increase efficiency for photometric (colorimetric and enzymatic) and electrochemical (pH and conductivity) analyses, discrete cell technology allows labs to measure multiple analytes simultaneously. The analyzers not only improve the reliability of results compared to manual techniques, they also increase laboratory productivity by freeing staff to walk away and work on other tasks.



Soil samples for analysis. Photo courtesy of Frind Lab Services.

Soil testing workflow

Sample preparation

After the client collects and delivers the soil sample to the lab, it is sieved to remove rocks and vegetation. Next the sample is divided into two parts, one to be dried and one kept wet to meet the requirements of the extraction methods that will be used. The sample pH is measured to provide insight about which extraction method will provide the best results.

Sample extraction

The specific extraction methods used are dictated by the analyte to be measured (Table 1) and are the industry and regionally standard approaches. The soils in BC are generally of high pH so the Olsen P Method would typically be preferred for phosphorus determination, however, Frind Lab Services uses both the Olsen P and Bray 1 methods for extractions because those are the methods with which clients are familiar. Though other methods may be easier, more efficient, or more compatible with the Gallery discrete analyzer, the Olsen P and Bray 1 methods produce results comparable to the results provided to clients in the past, including those obtained using other labs and other instruments such as segmented and flow injection analyzers. Tara Holitzki, Lab Manager, Frind Lab Services of Frind Estate Winery, noted "Truog extraction, which I used in the U.S., would be easier to set up in the Gallery discrete analyzer, but nobody seems to use it in Canada. Extraction methods are typically region-specific because of soil pH and other physical properties. We plan on setting up the Kelowna Method for phosphorus, which is a better extraction method for our region."

> "For the Gallery discrete analyzer, we went with the extraction methods already used in our region because farmers are familiar with those. They can look at our reports and know how to convert the results into fertilizer recommendations. There are other extractions we could use, for phosphorus for example, that I would prefer because they would be easier to analyze, but farmers anticipate the Olsen and the Bray methods."
>
> —Tara Holitzki

Table 1. Soil extraction methods

Analyte	Extraction method	Considerations
Phosphorus	Olsen P[1-5], Bray 1[6-9], and Kelowna[10-12]	Bray 1 method provides better results for acidic soils (below pH 6.5). Olsen P method provides better results for alkaline (above pH 6.5). Kelowna method is preferred for Western soils, has a large range for soil pH.
Nitrate and ammonium	2M KCl[13,14]	
Sulfate	Calcium phosphate[15-17], calcium chloride[17]	
Chloride	Calcium nitrate[18]	

Centrifugation, pH adjustment, and dilution

Following extraction, samples are filtered according to the method requirements. Unlike water samples, extracted soil samples also typically require pH adjustment and dilution to allow colorimetric measurement in the Gallery discrete analyzer. These steps minimize matrix interferences and prevent overwhelming the buffer capacity of the Thermo Scientific™ Gallery™ System Reagents, which would cause bubbling and precipitation, leading to poor results. Instead of water, extraction reagents are used for all dilutions (matrix matching) to avoid unexpected interferences.

When using the Olsen P extraction method in particular, the pH of the sample extract (which is about 8.5) needs to be amended with acid. Frind Lab Services observed that if the pH isn't adjusted, the Gallery reagents (pH 2 to 3) dissolve the extract and create bubbles, preventing reliable calibration curve development and results reproducibility. To perform the pH adjustment, the lab prepares the diluent by adding 5 N sulfuric acid to a tenfold-diluted Olsen P extracting solution at a ratio of 3 mL acid/100mL diluted extracting solution, and let it stand until there are no bubbles in the solution.

The Gallery discrete analyzer automatically performs the twofold dilution that is applied to the Olsen P extracts, which minimizes the sample preparation time and speeds sample processing. Holitzki explained "We add the dilution factor in the software interface and the Gallery discrete analyzer recognizes the diluent that's been loaded in the instrument as a reagent. The diluent and sample are then accurately transferred to the cuvettes for dilution which adjusts the sample pH before the Gallery system reagents are added for phosphorous determination. We get consistent duplicates, and the calibration curve looks really good (Figure 1). I just make sure that there is a lot of the diluent, and the Gallery discrete analyzer does everything for us." Chemical analyses are carried out automatically after the extracted samples, diluents and the ready-to-use Gallery reagents are loaded, and the method selected. The user can then walk away to focus on other important tasks in the laboratory while the analyzer performs analyses.

> "I like that the Gallery discrete analyzer dilutes your calibration curve. With the segmented flow and the flow injection analyzers, you have to make a calibration curve on your own, by serially diluting a stock solution. The Gallery makes calibration curves more accurate and allows for less human error."
>
> —Tara Holitzki

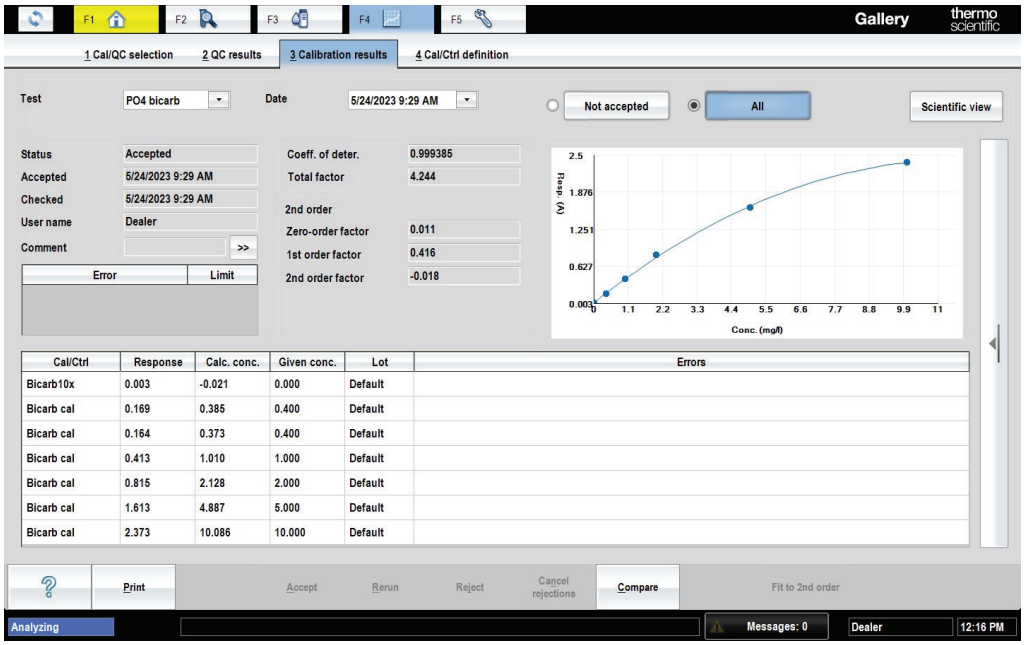


Figure 1. Gallery discrete analyzer calibration curve for Olsen P sample analysis

QC samples provide confidence in results

The lab uses QC samples to assess the performance of the extraction and measurement system. The QC sample is a lab-collected soil sample that was sent to four different labs across Canada for reference results. Holitzki explained, "our reference soil is extracted along with client test samples as a QC sample because I know what the values should be after each extraction. That's how we verify our extraction methods are accurate. There are many interferences in the soil extracts so it's important to make sure that you're actually getting good results by using an internal QC that is extracted alongside the soil sample. We also use an instrument QC to make sure our calibration is accurate, but that does not ensure that our soil extracts are accurate."

Frind Lab Services' state-of-the-art laboratory with the Gallery discrete analyzer. Photo courtesy of Find Lab Services.

"With the Gallery discrete analyzer, I can run multiple different types of samples at once and add different tests whenever it is required. With the flow injection or segmented flow analyzer, there's generally just one line dedicated to a particular test, with only one filter."

—Tara Holitzki

Gallery discrete analyzer benefits

Analytical throughput and flexibility

Frind Lab Services realizes notable advantages when using the Gallery discrete analyzer for testing. Most notably, the analyzer's speed of analysis and walkaway productivity help the lab address its high sample loads and meet its 30-minute sample turnaround goal for routine measurements. As Holitzki described, "the Gallery is faster running samples compared to flow injection or segmented flow instruments. It depends on the method and the dilution, but if the sample needs a dilution, the Gallery is about 2 to 4 times faster." Holitzki added, "I found that when I was using the segmented flow analyzer, I had to monitor the sample analysis and review the chromatograms while it was running to ensure that there were no interferences, clogs, or dilutions required for each sample. A lot more time was involved in a sample analysis run. The Gallery discrete analyzer allows you to load up your trays and request whatever tests you would like on each sample. The reagents are bought premade and if there is a calibration needed, it does not take a lot of time to add it. If a dilution is needed, then the Gallery will select a dilution based on the methods built into the software and automatically run them for you."

Some analyses require immediate results. For example, because free SO_2 (sulfur dioxide) prevents oxidation and suppresses unwanted microbial activity, producers need quick determination of the free SO_2 concentration in their wine prior to bottling. Using the Gallery discrete analyzer, the lab "can run the sample in three minutes and call with results before the client is back at their winery. Then they can adjust their wine accordingly," said Holitzki.

Frind Lab Services offers much more than soil sample testing, which is why it's important that the Gallery discrete analyzer can simultaneously measure multiple analytes in a variety of sample types including juice, wine, cider, and waters. Often labs must have a dedicated instrument for each test. According to Holitzki, "the Gallery is efficient because we can run analyses on wine, juice, berries, and soil at the same time. With other instruments you must have a channel setup for one analyte—nitrate for example—and you generally don't run anything but nitrate on that channel. Once you buy that instrument you buy it for running nitrate. With the Gallery you can change or add different tests at any time." The broad range of analyses that Frind Lab Services performs using the Gallery discrete analyzer are shown in Table 2.

Table 2. Analytes per sample type measured using the Gallery discrete analyzer

Soil	Juice and fruit samples	Wine and cider samples	Wastewater samples
Sulfate	Glucose	Free sulfur dioxide	Total Oxidized Nitrogen (TON)
Nitrate	Fructose	Total sulfur dioxide	Phosphate
Ammonium	Malic acid	Acetic acid	Chloride
Chloride	Nitrogen by o-phthaldialdehyde assay (NOPA)	Tartaric acid	Ammonia
Phosphorus	Free sulfur dioxide	D-glucose	Alkalinity
	Total sulfur dioxide	D-fructose	Fluoride
	Acetic acid	Malic acid	Sulfate
	Glucose/fructose/sucrose	NOPA	Urea
		Sucrose	
		Chloride	
		Total polyphenols	
		Glycerol	
		Lactic acid	
		Urea	
		Citric acid	
		Acetaldehyde	
		Gluconic acid	

"With the Gallery discrete analyzer, you don't need to be a chemist or an analytical scientist. You can easily teach others to use it because all they have to do is add the purchased reagents from Thermo Fisher Scientific, make a calibrator, and pour samples into the cup. You don't have to do anything else until the Gallery discrete analyzer tells you that the rack is done."

—Tara Holitzki

Loading samples and reagents into the Gallery discrete analyzer. Photo courtesy of Frind Lab Services.

Ease of operation

Ease of use is another important benefit of the instrument. Holitzki explained, "methods are very easy to build, and they are pre-loaded in the software, so you just have to make some adjustments to them here and there. Once the method is set up, it's very easy to load a rack of samples, make the calibrator and you're ready to run. I only need to make the calibrator stock solution because we buy the Gallery system reagents. The Gallery discrete analyzer dilutes the calibrator for the calibration curve." Easy operation adds confidence in analytical results. Holitzki added, "I trust the results produced by the Gallery. It's hard to have errors when all you need to do is fill a sample cup."

Adding to its ease of use, the Gallery discrete analyzer minimizes maintenance. "I like that there's not a whole lot of maintenance for the Gallery. With other instruments you usually need to change the pump tubing, and replace heaters, bulbs, and cadmium coils. There is nothing like that on the Gallery," said Holitzki.

Future interests

In the future, Frind Lab Services is interested in setting up the TON vanadium method to replace the TON enzymatic method for soil sample testing on the Gallery discrete analyzer. The lab determined that the TON enzymatic isn't feasible for use with the Gallery discrete analyzer when testing numerous samples due to the time required for reagent preparation and subsequent short reagent shelf life of eight hours. The TON vanadium medium offers the additional advantage of enhanced sensitivity.

About Tara Holitzki

Tara Holitzki, laboratory manager at Frind Estate Winery, has over 16 years of experience as a technician and later as an analytical laboratory manager preparing safety documentation, assisting students and researchers, conducting research, troubleshooting, maintaining and repairing equipment, purchasing lab consumables, developing new methods on instruments to fit the needs of the lab, assisting in grant writing to obtain new instrumentation, and writing reports and invoices for clients. She has set up and developed two fee-for-service labs including Frind Lab Services. Holitzki has extensive knowledge covering a wide array of sample preparation, analysis, and results interpretation approaches. In addition, she has a background in soil, water, plant and isotope processing, environmental and climate related research, and is using her previous experience to help Frind Lab Services serve BC with a state-of-the-art laboratory. Holitzki has a Master of Science (MS) in Tropical Conservation Biology and Environmental Science from University of HI, Hilo.

About Frind Lab Services

Frind Lab Services is a VQA-certified lab that uses state-of-the-art technology to provide an extensive variety of tests with quick turnaround times to support the needs of wineries, breweries, cideries, farmers, orchardists, and researchers. The lab offers individual and panels of tests including soil and plant testing for nutrient and heavy metal levels, pH and moisture; irrigation water testing for dissolved solids, pH, and chemical composition; fruit maturity testing including brix, glucose, fructose, malic acid, pH and total acidity to help managers make informed harvest decisions; fermented beverage testing including alcohol, sugars, free and total sulfur dioxide, and acidity to support production processes. Through wine cooperatives, the lab also supports student learning covering wine and instrumentation. Tests are performed using advanced analytical techniques including elemental and discrete analysis, Fourier transform infrared (FTIR) spectroscopy, gas chromatography tandem mass spectrometry (GC-MS/MS), inductively coupled-plasma optical emission spectroscopy (ICP-OES), flash combustion and UV–visible (UV-Vis) spectrophotometry.

Frind Lab Services is a part of Frind Estate Winery (https://www.frindwinery.com), situated on the historic Bennett Property in West Kelowna, BC. The winery is committed to lead through innovation and technology to create smooth, fruit forward wines and to providing a world class experience to guests with familial hospitality.

Method references

Olsen P method

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