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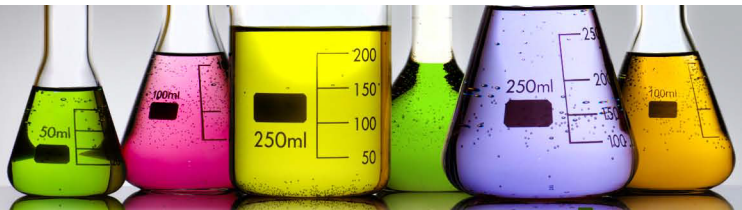
Committed to Quality and Service



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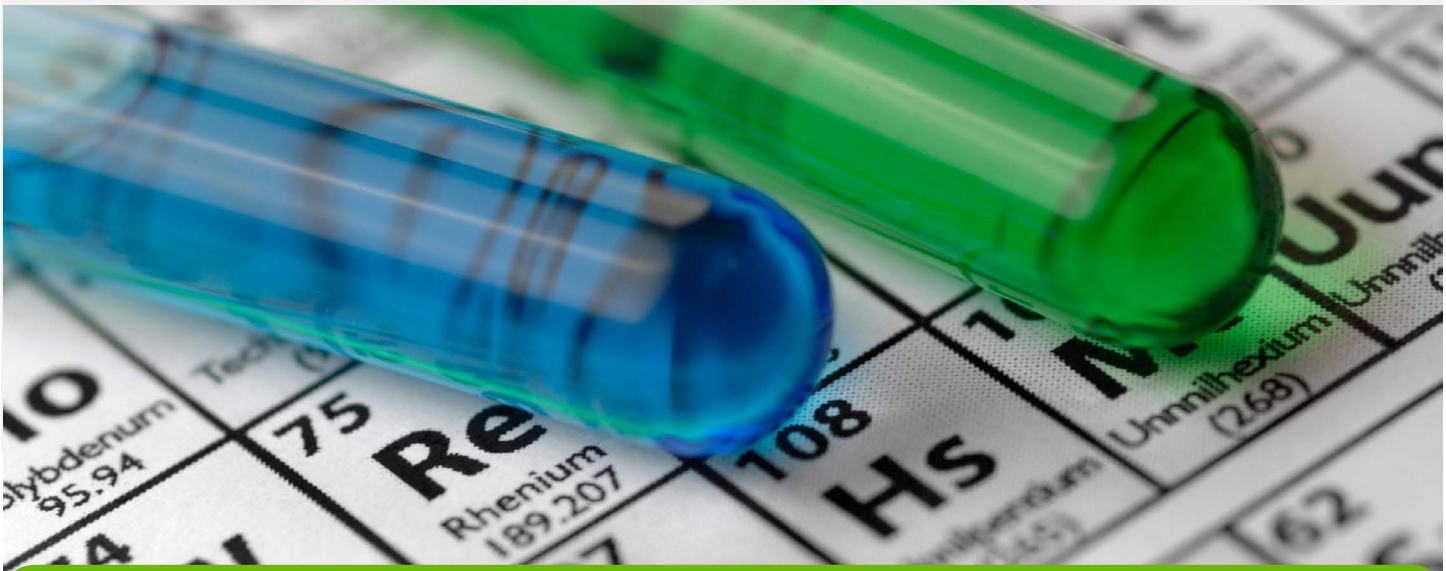
Assessing the Quality of Lab Data:
GETTING BACK TO BASICS



Science is an ever-changing landscape. For us in the environmental lab business, we've witnessed first-hand the huge advances made in the tools of our trade over the past few decades. The new generation of analytical equipment and computer/instrument interfaces make it hard to imagine that there ever was a day when technicians would stand by their instruments and manually inject samples, or manually titrate samples to an end point. Automation and computerization have certainly taken over. Follow that notion too far though and it may have one mistakenly believing that this implies better numbers and better data. It also suggests that the end-user can reduce their due diligence when reviewing lab data, when in fact nothing could be further from the truth.

in this issue

The following illustrates why you – the consumer of environmental lab data – need to understand that the scientific pillars of Precision and Accuracy are something that instrumentation re-engineering and sample processing speed might not necessarily account for. Increasingly the onus rests with you, the consumer, to assess the quality of your results and the quality of your lab. Below are some items you should consider:

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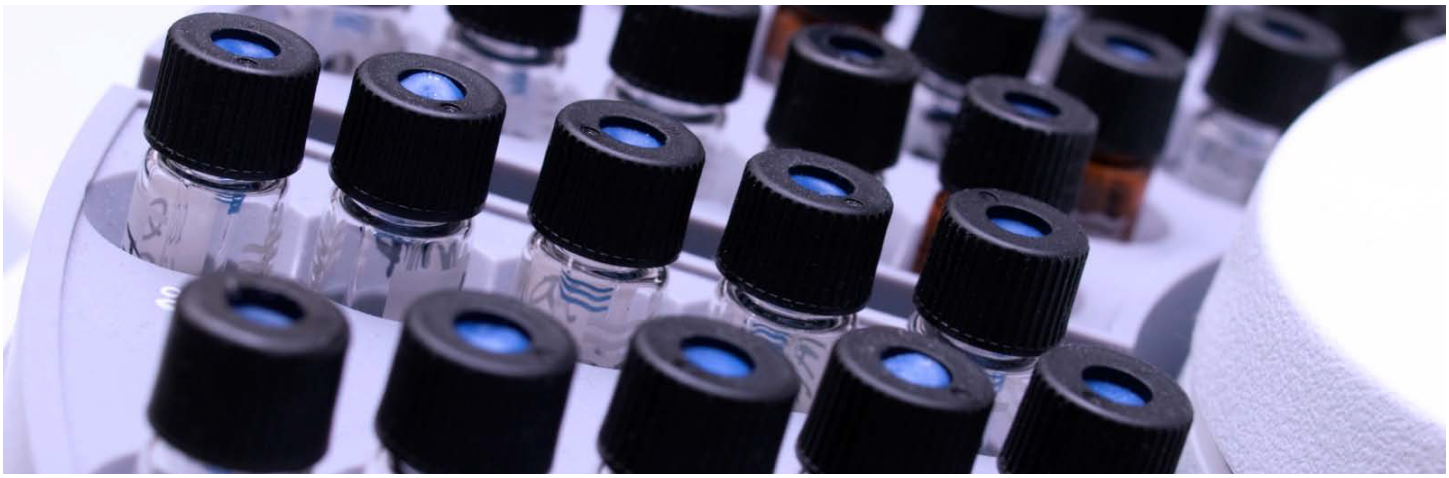

1 The Myth of Intelligent Instruments

No laboratory instrument is aware it is "doing chemistry". This may sound flippant, but if you grew up like I did watching 2001: A Space Odyssey or witnessing the advent of your parents' first dishwasher you likely got caught up in the romanticism that machines somehow know what they are doing. Lab instruments don't. They might be programmed to register wavelength or electrical impulse etc., but they certainly don't know chemistry. It takes many, many people-hours to set up, calibrate and validate a new piece of equipment. Everything from proper flow rates to sensitivities to computer interfaces needs to be considered. All these steps can introduce error along the way. Instrument manufacturers themselves aren't immune to errors as we've seen misprints in technical specifications, errors in calculations in software modules and faulty setup procedures reinforcing the notion that nothing is to be assumed correct until it is proven.

ISO 17025 – the international standard that environmental labs conform to – requires labs to validate instruments prior to implementation and undoubtedly this is a good start. However, as instruments frequently "drift" over time, a one-time validation on an instrument that pushes through hundreds of samples a day is not realistic. A good lab will routinely perform daily multipoint calibration checks on analytical instrumentation, and seek to re-validate from first principals in a timely manner. Extra effort beyond the ISO standard should be made, as you can't expect technology will self-diagnose or self-repair at any level. Recall your Grade 10 Chemistry teacher explaining the concept of accuracy versus precision – accuracy is how close the number is to the true number, precision is how repeatable the number is; just because an instrument spits out the same number on a sample again and again does not make it real. As a consumer of lab services, don't be shy to ask to see things such as the baseline run, quality control runs or the calibration curve of an instrument, and make sure it is from the timeframe related to your sample analysis.

2 Same Test Methods Can Yield Different Results

Analytical methods typically conform to an industry standard such as an ASTM, MOE, APHA or EPA standard protocol. Intuitively, we can suspect that these standard methods should ideally allow for apple-to-apple comparison of data. However, there are many interesting studies that point to cases where split samples sent to different labs using the same instrumentation and reference methods are statistically different. A wonderful article entitled "Trouble At the Lab" (The Economist, 2013) really brings this concept to life in citing research studies that failed to yield the same results when replicated step by step. So how can this occur? Assuming the sample was taken according to proper split-sample sampling techniques (i.e. there are no chemical differences inherent in the sample itself) there are still many other factors at play. For extremely time-sensitive tests such as formaldehyde, differences between Lab "A" prepping the sample 12 hours before Lab "B" can make a difference (even if the method hold time is met by both labs). Temperature fluctuations while in transit are another contributing factor. Method-to-method variance as well as same-method variance is an issue as well. Bench-level technique also plays a huge role. Just as a good surgeon will have better clinical results than a lesser-skilled surgeon, the same holds for laboratory staff. A well-trained, skilled Lab Analyst is key to ensuring accuracy and precision are apparent in the final number. Everything from having an organized and functional work station, excellent bench-level technique and an eye for detail are major contributing factors. We speak of "good hands" in this industry too and we don't refer to it lightly.

ISO 17025 does require that accredited labs participate in a Proficiency Testing Program twice a year for each accredited method. Results must fall within an acceptable result range or the lab will have that test suspended from their scope of accreditation (double failures will mean a suspension). Again, this provides some reassurance, but unfortunately when something as dear as accreditation is at stake, the consumer must not rely too heavily on the lab's PT track record as you can bet the lab ran and reran that sample with many extra quality control measures in place. It was likely not run as part of the day's normal production. A conscientious consumer of lab services can help identify weak data by blindly testing their lab with the inclusion of spiked samples. Knowing the experience and qualifications of the analysts running your sample is also something you should feel comfortable discussing with your lab.

3 The Need for Speed

The focus on the "need for speed" in the analytical testing industry undoubtedly comes at a cost. If doing it fast and cheap are the drivers, then expect that equipment maintenance, staff training and adherence to best-practices in analytical chemistry will start to slide. Some corners that might be cut are things as fundamental as labs failing to run multiple dilutions for samples – a key step in ensuring the result range is optimized for the calibration of the instrument. Other notables are cutting out critical steps in standard methods such not running the accompanying moisture test for an organic analysis of soil (e.g. BTEX, F1-F4, etc.) which undoubtedly saves time, but can seriously underestimate the concentration of the contaminant in the sample and can grossly misrepresent the true method detection level of the sample. Again, as a consumer, simply asking the lab to show you their dilution runs or the moisture calculation for your soil sample is enough to give you confidence that you are doing your part in assessing the quality of your data and your lab.

CONSUMER'S TOOL KIT FOR ASSESSING DATA QUALITY AND LAB PERFORMANCE:

Beyond some of the suggestions mentioned above, other tools the consumer can use to assess the quality of the work performed by their lab include:

- Make the most out of incorporating field and trip blanks into your cooler orders and send the occasional blind sample duplicate as well;
- Assess whether your lab is using additional Quality Control measures beyond control standards and duplicate analyses in their batches. Things to look for include the use of control charting in the lab to flag statistical variation in a method over time or the inclusion of matrix-specific Certified Reference Materials;
- Ask your lab to define what a "batch" of samples is for them in terms of "we run these quality control measures per batch" (i.e. is it every 20 samples, every 100, once a day, once per instrument run?). An average batch size shouldn't exceed approximately 20 samples per test;
- Understand the concept of statistics in your data when assessing your data set. If you aren't certain, take a refresher on the concept of allowable variance and relative percent difference (RPD) between two numbers before deciding that they are too different. Also understand that RPD varies by analytical method and by matrix (soil versus water) to allow for known errors;
- Understand the concept of Method Detection Limit when assessing your data set. If a lab reports a blanket Detection Limit for an analysis, you must consider that the actual detection level (i.e. how low the lab can detect/report for that analyte in that particular sample) may be disappointingly above that. In the worst case, it may be so far above the blanket Detection Limit (sometimes referred to as a Reportable Detection Limit) that the result may not pass your regulatory criteria you are comparing to. Remember, less than detect is only valid if you know what the "detect" really is.

Overall, the onus to provide quality data rests with the lab. However, in the era of automated high-throughput and price erosion, the onus to assess the quality of your lab rests with you.

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Sound Environmental Sampling Techniques Translate into Better Lab Data:

FIVE SAMPLING PITFALLS TO AVOID

As an environmental testing lab, we see many environmental samples a day - some for ongoing monitoring purposes, some strictly for compliance reasons, and some for investigative work. We also appreciate that the cost associated with collecting these samples often goes far beyond the cost of the analytical tests themselves when you factor in expenses associated with trained field staff, sampling equipment, transportation to/from remote site locations etc. Therefore, it concerns us when we receive samples at our various labs that contain inconsistencies and non-conformances, particularly knowing that some of these pitfalls will undoubtedly affect the quality of the resulting lab data.

If big decisions rest on the numbers coming from your environmental lab, then please read on to learn how the top five sampling pitfalls can impact your results.

Pitfall 1 IMPROPER USE OF SAMPLING CONTAINERS

One of the first pitfalls to avoid concerns the proper use of your sampling containers. This may seem basic enough, but it is surprising how many times we in the lab industry see sample submissions that fall drastically short in this respect. When you set out to sample, you've undoubtedly discussed your sampling program with your analytical lab ahead of time to ensure you have the right bottle sets for the job. It may appear excessive to you when you flip open the lid of your sampling cooler and find a myriad of glass and plastic bottles sent from the lab for what you feel is a pretty straightforward sample collection plan, but the choice of sampling containers has been well-considered by your lab and should not be second-guessed. For example, organics love to cling to plastics, which is why glass bottles with Teflon-lined septa are generally used for samples that will be analyzed for organic compounds. Light is also a destructive force for some analytes which is why amber glass is provided, or bottle forms are used to protect the sample from the effects of light degradation for certain analytical tests.

As for the plastics, a closer look will reveal they aren't all the same. Testmark provides PET plastics for general chemistry parameters, higher-grade HDPE bottles for nutrient analysis, and acid-rinsed HDPE bottles for metals analysis after extensive in-house testing confirmed that trace amounts of silicon, aluminum, titanium, magnesium, chromium, zinc and iron can leach out of the HDPE plastic and into your sample if not acid-rinsed first.

Then there is the issue of volume. If the lab provides a set of bottles, please use them – all of them. The ability of your lab to achieve a low method detection level (MDL) is as much a function of the methodology and analytical equipment used as it is the volume of the sample. Bottles returned only half-filled run the risk of yielding a higher MDL as MDL is inversely proportional to sample volume.

Finally, it is important to consider the proper use of preservatives. Again, this is something you can request from the lab when placing your bottle order (pre-charged sample bottles) which can drastically prolong the sample hold time as well as improve the quality of the data itself. Preservatives, similar to bottles, are analyte-specific. In general, preservatives serve the purpose of stabilizing the chemistry by either reducing the biological activity in the sample after it is collected, neutralizing other chemicals such as chlorine, or in some cases trapping the analyte of interest to prevent loss to head-space. Pre-charged bottles have specific quantities of preservative (generally an acid or a base) in them and are fit for purpose. Field errors such as overfilling a pre-charged bottle essentially dilute the effect of the preservative. Another common error is submitting preserved samples for lab filtration and analysis as it is flawed to filter samples after preservation.

Pitfall 2 IMPROPER SAMPLING TECHNIQUE FOR THE ANALYSIS OF VOLATILES

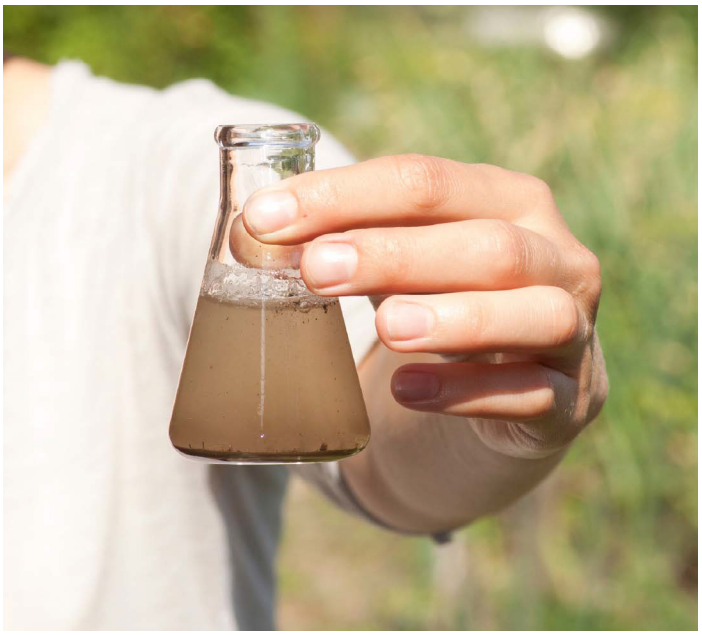
The amount of headspace in water and soil samples can often greatly affect the quality of your lab data. For example, samples collected for the analysis of volatile organics, THMs, dissolved oxygen and suphide should not contain headspace. The goal is to fill the bottle just enough to achieve a positive meniscus and then seal it with the lid. A quick field check means flipping your bottle over and seeing if there are any visible air bubbles in your sample. An air bubble that covers the entire surface of the bottom of a 40mL vial (used for the analysis of BTEX/VOC, F1 and THMs) is too much. Volatile compounds, by their very nature basically want to 'be air' which means you are essentially losing some of your target compounds to headspace.

Particular attention has been given to the case of volatile organics in soil in light of the amended Brownfields Regulation. Best practice involves the use of either hermetically sealed sampling kits, or the more convenient methanol pre-charged soil sampling kits. Testmark promotes the use of the latter and provides Terracore® samplers to our clients which allow them to extract two soil plugs and insert them in pre-charged methanol vials. The methanol serves to "trap" the volatile compounds in solution until they can be sparged at the lab.

Pitfall 3 EXCESSIVE PARTICULATE MATTER IN WATER SAMPLES

This is a tricky one to avoid given some of the realities that exist when drawing from silt-laden or under-developed groundwater monitoring wells. We often hear from clients about the logistical challenges of filtering samples in the field when there are time constraints or they are working in remote locations. At times, the only viable solution may be to collect the sample as is (no filtration) and get it to the lab as soon as possible to be filtered there prior to analysis.

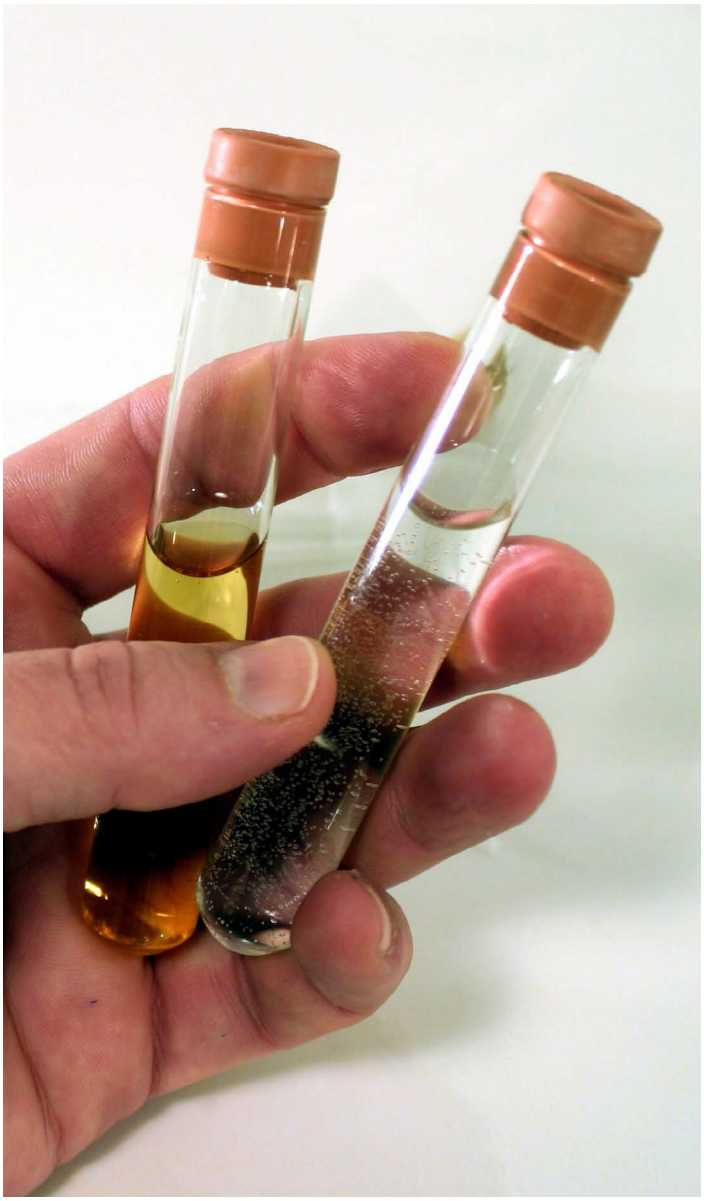
It is challenging to think of any analytical test that can't be affected by the presence of sediment in the sample. The presence of suspended particulate can greatly affect almost all microbiological, nutrient or organic tests results. Take the example of polyaromatic hydrocarbons (PAH) analysis where PAHs tend to adsorb to particulate to the extent that samples with visible sediment may be biased high. The revised Record of Site Condition provides some accommodation for this in the form of qualifiers for filtered and unfiltered PAH samples and also underscores that the best practice for metals analysis is to field filter.



The case of metals is also greatly affected by the presence of sediment and here we get into the realm of colloidal chemistry, which can be seen almost as a science unto itself. Colloidal and particulate metal may be found in a number of complexes including hydroxides, oxides, silicates and sulfides. Furthermore, metals in water are continually adsorbing and desorbing to and from sediment; they are in a constant state of flux dependent largely in part on the water chemistry. Adsorption removes the metal from the water column and stores it in the substrate. Desorption returns the metal to the water column. Metals may be desorbed from the sediment from such things as increases in salinity, decreases in redox potential or decreases in pH. Dissolved metals are generally in low concentrations in natural water bodies. The bottom line is that particulate matter in your sample can give fodder to all these biases.

Pitfall 4 FAILURE TO INCORPORATE QUALITY CONTROL MEASURES IN YOUR SAMPLING PLAN

It's often been said that North Americans have become the most over-insured society on the planet. We have been known to insure our health, lives, cars, homes, vacations, kids, pets, even body parts - pretty much everything. But surprisingly, when we function at our jobs, it is remarkable just how insurance-adverse we are. A properly executed sampling plan should include some form of quality assurance and quality control. Think of it this way - for the inconvenience of a few extra bottle sets, you have some assurance that your sampling design, your field work and your choice of lab is validated.



The US Environmental Protection Agency (EPA) has a good number of guidance documents that address how to incorporate critical QA/QC measures into your sampling plan. For example, they recommend one trip blank per cooler. A trip blank is a clean matrix sample (lab-grade water) taken from the lab to the sampling site and transported back to the lab without having been exposed to sampling procedures. Field blanks are analyte-free water (lab-grade water) that is poured and preserved in the field exactly as if it were an actual field sample. Where trip blanks are meant to provide QA for the quality of the bottles and denote any lab-based contamination, field blanks go one step further by also providing a QA check on the physical sampling technique used in the field. The EPA recommends the use of 1 field blank/day/matrix or 1 blank/20 samples/matrix (whichever is more frequent).

Other insurance you should consider building into your sampling plan includes taking field duplicates or splitting samples between labs. If you opt to include these elements, just be sure your sampling technique is valid to ensure you aren't introducing sample bias by, for example, taking consecutive samples as opposed to splitting a homogenized sample.

Finally, a major QA/QC consideration when dealing with environmental chemistry is to ensure you are transporting samples with due regard to temperature and hold-time requirements. Your lab should provide you with guidance in regards to sample hold-times as they do vary according to test method. In general, protocols favour that samples be maintained between 2-8°C during transport.

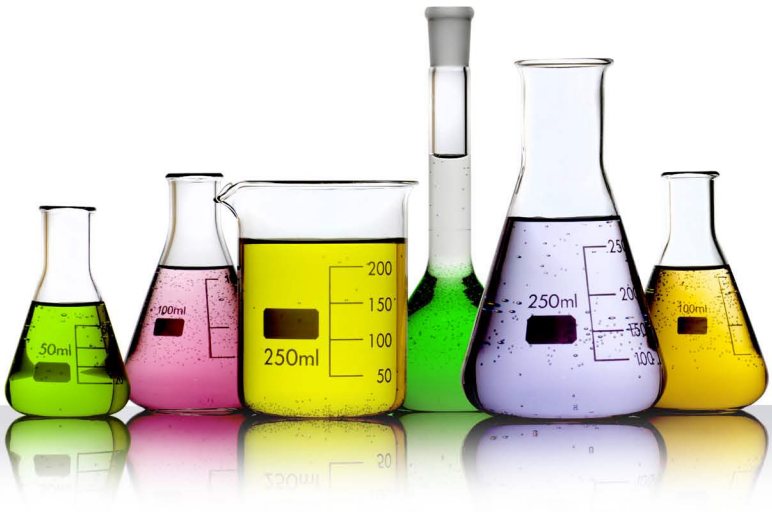
Pitfall 5 POORLY DEVELOPED SAMPLING PLANS

Sampling is an arena that Testmark doesn't enter into firsthand. We consider our core business to be the provision of quality analytical testing services and we stick to that. However, we are extremely aware of the impact sloppy sampling regimes will have on the interpretation of lab data. Once again, the US EPA is a great resource to turn to as they have developed extensive guidance documents in regards to how to carry out water, soil, sediment, biota and air sampling programs. All factors must be considered, from the influence of stratified chemical and thermal layers occurring in natural water bodies, to best practices in sinking and developing groundwater wells. Statistical representation in terms of number of samples and spatial distribution is also critical.

From a lab perspective, we experience a similar situation when we perform extractions for organic analyses. Extractable organics typically require 1 litre of sample to be submitted to the lab. However, the instrument (gas chromatography) requires only about 2mL of an extracted aliquot. The key then becomes ensuring that the 1 litre sample is extracted representatively to ensure that the 2mL aliquot contains any compounds of interest. This involves sound adherence to extraction protocols (be it solvent extraction, Soxhalet extraction etc.).

The end goal is that the sample is meant to represent the whole. A sound sampling plan should consider the impact of all variables including ones such as statistics, time, temperature, seasonality, numeracy and location.

Undoubtedly, there are other pitfalls beyond the ones mentioned above that can impact the quality of your lab data. However, the main message here is to take the time beforehand to properly prepare and execute your sampling plan and to learn as much as you can in regards to best practices for sampling. Time spent upfront at the planning stage, as well as care taken in the field, can translate into much more meaningful results when the data comes back from the lab. Consider your lab your partner throughout the process. Much of the technical direction concerning the proper use of laboratory bottles, hold times and preservatives is information that they will readily provide.



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