



LABNOTES

News & Updates of the LabCert Program



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In our quest to get back on track with publishing TWO editions of LabNotes each year, here is our second issue for 2015. There's a lot of helpful detail regarding ICP interference correction plus many more articles that should prove both timely and helpful. Enjoy! But please remember to share this with others in the lab; our system supports only a single e-mail contact per lab.

For ICP: Move On From ICS-AB – It's Time

Parting with something we have clung to so tightly is stressful. Remember having to retire that "blanky" or well-worn, matted, and malodorous stuffed animal from one of your children? It's also true that we cannot reach for something new when our hands are full of yesteryear's junk. And so it is time for us all to part ways with our trusty friend, ICS-AB, that cherished vehicle so often (and incorrectly) used by labs nationwide to demonstrate that all spectral interferences have been properly addressed.

It's hard to believe that it's been 35 years since the CERCLA program, better known as the Superfund program, was enacted. The Superfund program spawned the EPA's Contract Lab Program (CLP), which served to coin the phrase "producing data of known and documented quality".

One thing the CLP program got right, that none of the more current and approved methods for ICP-OES technology included, was a clearly designed and articulated mechanism for evaluating spectral interference correction. Thus was born the concept of analyzing ICS-A (interferents only) and ICS-AB (interferents plus target analytes) samples. So popular (*and perhaps because protocols in methods such as 200.7 and 6010 were virtually impossible to decipher*) was the CLP's spectral interference correction verification protocol that it has been adopted by the vast majority of ICP labs in the country - even those that have never performed CLP testing.

ICS-AB acceptance criteria significantly mask interferences



At the risk of building it up just to crush it back down, the CLP approach was (and still is) a flawed protocol. The original acceptance criteria ($\pm 20\%$ of true value for all spiked analytes) allows spectral interferences of 200 ppb or more to go undetected! When routine axial detection limits for many analytes are below 10 ppb, that's a significant flaw.

ICS-AB was designed for pre-1985 instruments!

As if being flawed wasn't enough of a reason to move on, the approved method themselves built in a sunset clause for the use of good old "ICS-AB". Both the currently approved EPA method 200.7 (7.13.6) and 6010D (4.5) clearly state that, "*If the instrument does not display negative values, fortify the SIC check solution with the elements of interest...*". SIC (spectral interference check) is just a re-arrangement of the CLP acronym, ICS. And every functioning ICP today has the ability to display negative values, therefore no one should be spiking target elements into interference check samples!

As Bryan Brown's character in the movie, "Cocktail" so eloquently stated, "Coughlin's Law: bury the dead, they stink up the joint." It's time to bury the concept of ICS-AB.



New Auto-Calculating Benchsheets

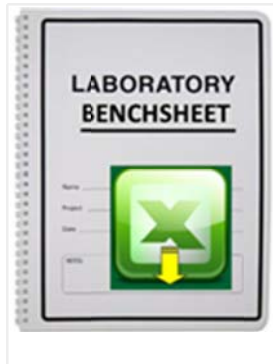
... these will not only provide accurate calculations, they will highlight QC failures

The LabCert team has put together updated, standardized benchsheets for BOD, TSS, Total Phosphorus, and Ammonia (both ISE and Colorimetry). These benchsheets can be printed off from a PDF version and used to document your results as they are. Alternatively, the Excel benchsheets can be used to calculate your results! Simply provide the raw data, and the benchsheet will not only provide accurate calculations, they will also notify you of QC failures and provide helpful feedback if your data doesn't seem quite right. Things like DO supersaturation, the absorbance of a phosphorous sample dilution over the absorbance of the high standard, and TSS reporting requirements will all be made clear by the interactive benchsheet. It's almost like having an auditor right there in the lab with you! In addition to the benefits of auto-calculation and highlighting QC failures, standardized benchsheets have the benefits of:

- Improved analyst satisfaction
- Improved lab/auditor communication and collaboration
- Increased efficiencies
- Enhanced data collection
- Opportunity to implement recommended practices (built-in)

The spreadsheets and PDF versions are available on [the LabCert website](#). Or individual benchsheets can be downloaded:

- [BOD benchsheet](#).
- [TSS benchsheet](#).
- [Ammonia by ISE benchsheet](#).
- [Ammonia by Colorimetry benchsheet](#).
- [Total Phosphorus benchsheet](#).



If you need any assistance, please contact Dave Ekern at david.ekern@wisconsin.gov or 608-785-6364.

Sample Preservation for colorimetric NH₃

... HACH method instructions conflict with federal and state rules

The LabCert program has encouraged labs to switch to the colorimetric method over the traditional ion selective electrode method as it is a better method and will save the



laboratory time and money. And now labs are applying to make the switch pretty regularly. There's just a wrinkle we need to smooth out to ensure that labs are in compliance with federal and state preservation requirements.

Most labs making the switch to colorimetry are using the Hach TNT plus Ammonia Method 10205. This method uses Test N Tube (TNT) vials and colorimetric technology to determine ammonia in water samples. TNT method 830 and 831 are both approved for wastewater and aqueous sample analyses. Note that TNT 832 is **not** similarly approved.

Here's the wrinkle: The complete TNT method written in proper EPA format (Method 10205) does not conform to NR 219 or 40 CFR Part 136 requirements with respect to the acid used to preserve samples (if they will not be run immediately).

The EPA formatted HACH method for TNT830 and TNT831 (10205) instructs the user to preserve the samples by adding hydrochloric acid (HCl):

Sample collection, preservation and storage

- Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.
- Preserve the samples by reducing the pH to 2 or less with at least 2 mL of **hydrochloric acid (HCl)**.
- Store at 4 °C (39 °F) or less.
- Preserved samples may be stored up to 28 days.

On the other hand, ch. NR 219, (Wis. Admin. Code) which is based on Clean Water Act

requirements in 40 CFR Part 136, requires that samples be preserved with sulfuric acid (H₂SO₄).

Table F (Continued)
Required Containers, Preservation Techniques, and Holding Times for wastewater

Parameter Number/Name	Container ¹	Preservation ^{2,3}	Maximum Holding Time ⁴
Table B — Inorganic Tests			
1. Acidity	P, FP, G	Cool, ≤6°C ¹⁸	14 days
2. Alkalinity	P, FP, G	Cool, ≤6°C ¹⁸	14 days
4. Ammonia	P, FP, G	Cool, ≤6°C ¹⁸ , H ₂ SO ₄ to pH=2	28 days

So...what is the correct approach? HCl or H₂SO₄?
In this case, the federal and state rules (NR 219) trump the method requirements.

40 CFR Part 136.6(b)(3) states,

"Restrictions. An analyst may not modify an approved analytical method for a method-defined analyte. In addition, an analyst may not modify an approved method if the modification would result in measurement of a different form or species of an analyte (e.g., a change to a metals digestion or total cyanide distillation). An analyst may also may not modify any sample preservation and/or holding time requirements an approved method."

Therefore you MUST use (sulfuric acid) H₂SO₄ to preserve samples, not HCl.



the fact that the EPA was changing its approach to how approved procedures from *Standard Methods for the Examination of Waters and Wastewater* (i.e., "Standard Methods") were cited in the Federal Register. They did just that; the EPA promulgated the changes to the Clean Water Act (40 CFR Part 136) in 2012, and on June 1, 2015, the changes took effect in chapter NR 219, the administrative code designated as a repository for approved analytical methods for wastewater analysis.

Gone is the concept of "editions" of Standard Methods. In fact, eventually there may not even be hard copy printings. From this point forward, the EPA will name Standard Methods procedures according to their year of approval by the associated Standard Methods committee. Of course, this change has only been made for the Clean Water Act to date; the EPA has not made this change for approved drinking water methods...at least not yet. The Federal Register and NR 219 now only cite approved Standard Methods procedures by their year of approval. Therefore, rather than being able to cite any one of the 18th, 19th, 20th, 21st editions or the online version of method "5210B" for BOD, the approved method must now be referenced as 5210B- 2001.

Seems pretty trivial...right? Wrong! Using and reporting approved method numbers is critical. For example, reporting an unapproved method reference for your PT testing results will require you to obtain a second PT sample. This is because PT results reported using an inappropriate method number or PT method code are not acceptable and will not be uploaded into our database. Similarly, labs are required to use—and follow—approved methods. Consequently, you will be cited during an on-site evaluation if you:



- Do not have a copy of the approved methods you reference on hand,
- Your SOPs have not been changed to reference the new Standard Methods naming convention, or
- If you do not report the method properly on your results.

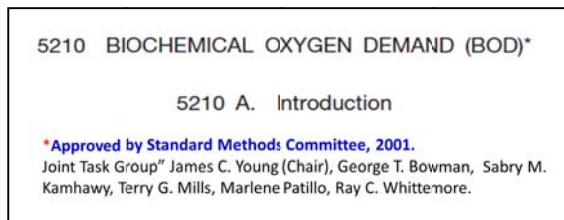
The Force Awakens - Standard Methods Naming Changes Take Effect



There has been a great disturbance in the Force...as if millions of voices suddenly cried out in terror and were suddenly silenced. Yeah...change can have that effect. A long time ago, in this very galaxy, we alerted you to

So...how do you know which "Standard Methods" method(s) are currently approved?

The approved date is found by checking the footnote on the method introduction (in hard copy editions), as illustrated below:



On-line method versions will always reflect the most current approved version. The approval date can be found on the Standard methods on-line subscription site (<http://www.standardmethods.org/Store/BrowseSM.cfm>).

Individual methods can also be purchased at a cost of \$69.00 each. Note that when you purchase a method, you purchase all of the letter designations for that method for that one price. For example, by purchasing 4500-NH3 (Ammonia), you get the ISE and the colorimetric procedures as well as all the other methods for ammonia.



Standard Methods has also developed a pair of cross-reference tables, one each for the Clean Water Act (wastewater) and the Safe Drinking Water Act (drinking water):

[Wastewater Approved Standard Methods link \(PDF\)](#)

[Drinking Water Approved Standard Methods link \(PDF\)](#)

For routine wastewater analyses, the following is a summary of the official approved version of the method as well as a list of editions in which the currently approved version is printed.

Test/Parameter	Approved Method	Equivalent to
BOD/cBOD	5210B-2001	21 st ed., On-line,
Ammonia (ISE)	4500-NH3 D-1997	20 th ed., 21 st ed., On-line
Ammonia (Color.)	4500-NH3 G-1997	20 th ed., 21 st ed., On-line
	4500-NH3 H-1997	20 th ed., 21 st ed., On-line
Total Phos. (manual)	4500-P E-1999	21 st ed., On-line
Total Phos. (auto.)	4500-P F-1999	21 st ed., On-line
TSS	2540 D-1997	20 th ed., 21 st ed., On-line

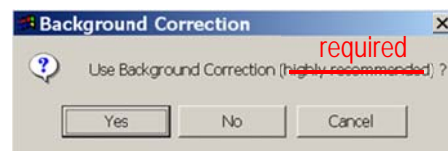
Notice that if you have only the 20th edition Standard Methods—or an **earlier edition**--, that's not going to cut it. Only the 21st edition or the online edition will cover all of the common wastewater tests. Even if you only do BOD and TSS testing, BOD requires the 21st edition or later. Note also that while Standard Methods suggests that the 22nd edition is also "approved", we cannot allow methods from that edition until it has been officially promulgated in the Federal Register. So we are essentially at the US EPA's mercy here.

Approved Method Codes for PTs

We are also providing a list of approved method codes for PT results. Note that if you use the State Laboratory of Hygiene (SLH) as your PT Provider, check the method code very carefully as their database retains the code used last which may not be correct. Be proactive and re-report the proper method code for all PT results.

Method Code	Method Name	Revision
BOD, cBOD		
20135006	SM 5210 B	21st ED
20135255	SM 5210 B-2001	
TSS		
20050800	SM 2540 D	20th ED
20051007	SM 2540 D	21st ED
20051201	SM 2540 D-1997	

Method Code	Method Name	Revision
Ammonia (ISE)		
20109200	SM 4500-NH3 D	21st ED
20109006	SM 4500-NH3 D	20th ED
20109404	SM 4500-NH3 D-1997	
Ammonia (Colorimetry)		
60005007	HACH 10205	5th ED
20111006	SM 4500-NH3 G	20th ED
20111200	SM 4500-NH3 G	21st ED
20111404	SM 4500-NH3 G-1997	
20111802	SM 4500-NH3 H	20th ED
20112009	SM 4500-NH3 H	21st ED
20112203	SM 4500-NH3 H-1997	
Total Phosphorus (manual)		
60003909	HACH 8190 (Equiv)	5th ED
60003896	HACH 8190	4th ED
20124009	SM 4500-P E	21st ED
20124214	SM 4500-P E-1999	
Total Phosphorus (automated)		
20124805	SM 4500-P F	21st ED
20125013	SM 4500-P F-1999	



Background correction is not the same as spectral correction

It is important to be clear that background correction is separate and distinct from spectral interference correction. In fact, if background correction is adjusted, then any spectral interelement correction (IEC) factors **must** be re-evaluated as well, while changes to spectral interference corrections (IECs) do not require adjustment of the background correction.

Background correction adjusts for changes in intensity unrelated to (although they can be affected by) spectral overlap. Spectral interference correction (SIC) is a separate correction made to adjust for emission intensity stemming from a neighboring target analyte or interferent element. Spectral interference correction historically results in the generation of one or more inter-element correction (IEC) factors. Because spectral overlap always ADDS to the emission intensity, IEC factors are negative adjustments (*based on the concentration of interferent, a portion of the emission intensity is subtracted from the gross intensity for an analyte of interest*). When background correction points are situated incorrectly—near an interferent—target peak integration can result in a negative intensity which must be compensated for by a positive spectral correction factor that adds back the area removed by the errant background correction point (see Fig. 6A below).

Background correction models

Modern instrumentation offers a number of ways to deal with background correction. These decisions are element and line specific. While most vendors establish “default” background correction for you, the point (or points!) where correction is made may not be suitable for the element and wavelength you choose to use. ICP is not “plug and play”; these default parameters need to be evaluated based on YOUR lab and the emission lines YOU choose.

Conventional, or “off-peak” background correction (OPBC) uses either one or two points adjacent to each target analyte. The point(s) selected for background correction needs to be

Background Correction in ICP-OES

The back-story on background correction

Background correction is a mechanism to compensate for variable background contribution to the determination of the analytes. Why is the background variable? The variability is an issue because quantitation is based on emission intensity and background intensities can change based on many factors, including changes in the plasma, changes in the argon supply/flow, changes in sample viscosity or acid concentration, and even room temperature/humidity. Therefore, to correctly measure the net signal intensity of a given sample for a specific element, we have to have a means of accurately determining the background intensity at the time of analysis in close proximity to the peak of interest.

Background correction: desired...or required?

For many analyses we focus our efforts on ensuring that labs are not adjusting sample results for background response (e.g. blank subtraction). ICP is quite the opposite. In fact, background correction is an absolute requirement of EPA methods 200.7(\$2.2) and 6010D (\$2.3)].

at a location that is not affected by other routine-- or even unexpected-- analytes.

We've seen many instrument default configurations that establish two points of correction at locations equidistant from and on either side of the target analyte peak maximum. ICP, however, is a case where two points are generally not better than one. The use of two background correction points should be reserved for those situations where an element peak appears at an area of sloping background (e.g., potassium).

In addition to conventional OPBC, instrument manufacturers now offer a number of unique systems based on proprietary algorithms (e.g. Agilent's FACT and FBC technologies; Varian's MSF system) to establish "fitted" background correction. At the risk of over-simplifying things, these systems analyze background intensity at various locations around each target peak and develop an algorithm which (theoretically) reflects true background, allowing the analyte peak to be properly integrated. In addition to background correction the FACT and MSF systems offer a means of spectral interference correction.

Setting background correction points appropriately

The position used should be as free as possible from spectral interference and should reflect the same change in background intensity as that which occurs at the analyte wavelength being measured. Note that while method language suggests that correction is made at a single location (*"The **position** selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line"*), technology has changed and there are advanced mathematical applications that provide improved correction by calculating a fitted background. These fitted background correction systems are acceptable for use even if the methods have yet to be updated to reflect as much.

Background correction -Examples

Figure 1 is a great example demonstrating how background intensity changes. This is a 6.25% Ca standard superimposed over a nitric acid blank. This is part of an excellent ICP reference offered

by Inorganic Ventures at <http://www.inorganicventures.com/icp-operations-guide>.

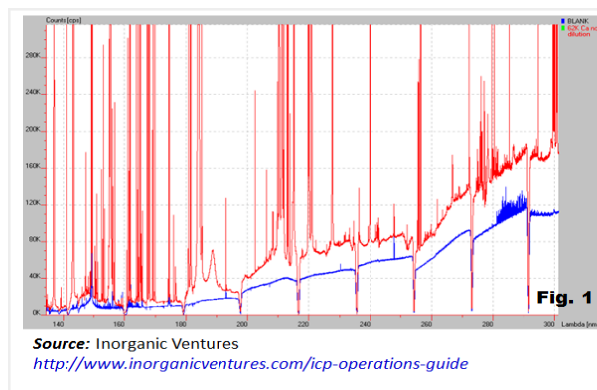


Figure 2 is an example of improperly placed background correction points. Not only are two points used on a non-sloping background, but both locations are contained within the wings of the analyte peak itself. Using these as background correction points will result in low bias for the peak integration.

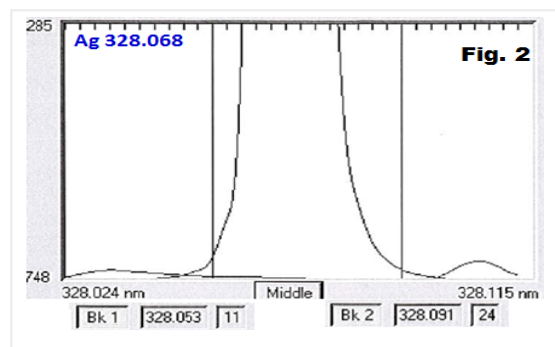


Figure 3 illustrates a case where two background points is more appropriate. In the region of shorter wavelengths, it's not uncommon to have a sloping baseline. Sometimes analyte peaks can appear as a peak coming off of wing overlap from elevated levels of an adjacent analyte. Using two points in these situations helps ensure accurate peak integration.

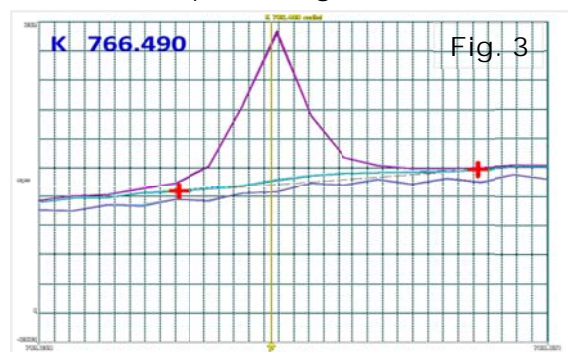


Figure 4 is an example of a case where a single background correction point is appropriate, and one is really forced to place it on the shorter wavelength side (point A). Selection of point B for background correction would be a problem if the sample contained copper (typical) or titanium (atypical).

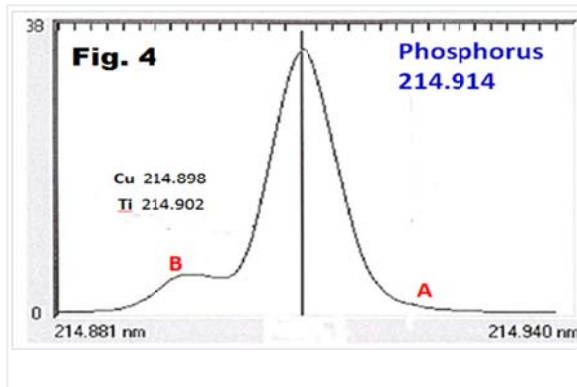
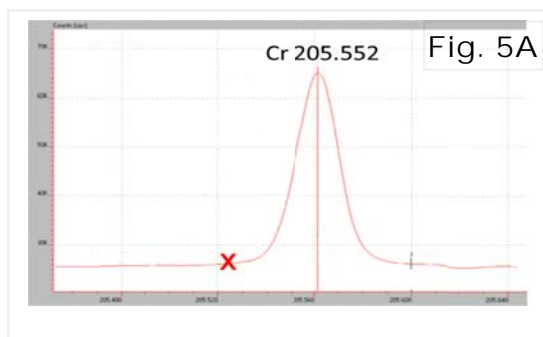
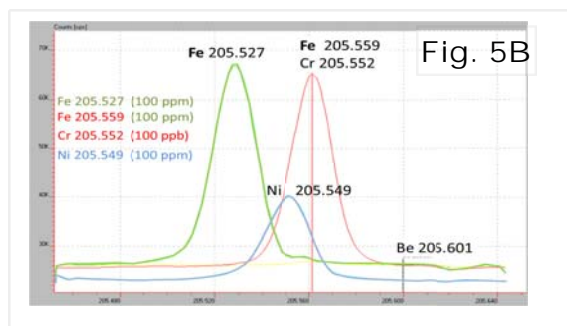


Figure 5A seems, at first glance like a no-brainer. Either side looks clear (from the single element standard view!). While either side of the target peaks appears to be appropriate for background correction (based on this scan), a decision is made to make background correction on the longer wavelength side. And that's why we have to consider what else COULD appear (and impact background correction).



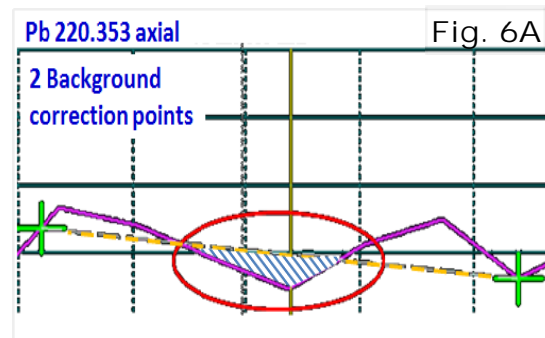
Unless we look at how alternate lines associated with other target analytes impact integration, however, a poor choice could be made with respect to OPBC. Fig. 5B shows that bigger picture.



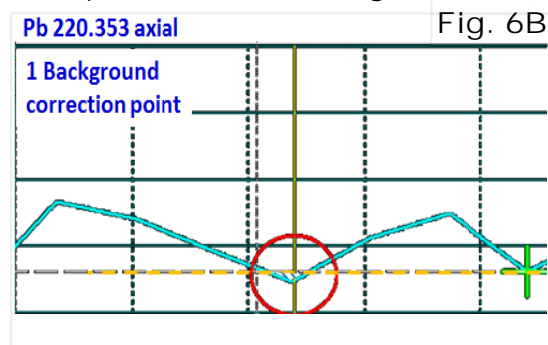
While there's a possibility of Be impacting the short wavelength side, Be is not typically found at high levels, and thus this is likely the better choice due to the traffic jam of wavelengths appearing on the longer wavelength side (Fe, Ni). In fact, there's a line for Fe that virtually overlaps the Cr peak, and Ni is almost obscured beneath Cr. This explains why 267.716 is the preferred wavelength in method 6010D. The currently promulgated version of method 200.7 recommends using this 205.552 line, but 200.7 has not been updated since 1994. Ultimately, as illustrated in figure 5B, 205.552 is not the best choice of wavelength for Cr.

Figures 6A and 6B illustrate the effects of 1 versus 2 background correction points when also affected by a challenging matrix and adjacent spectral overlap.

In Fig. 6A, there is Pb present in the sample at approximately 40 ppb. As the integration using 2 background correction points shows, one point is affected by an interference resulting in negative integration (and thus a negative result for Pb). In this case, a significant positive interelement correction (IEC) was required (which the lab had properly identified and established).



When the analyst removed the affected background correction point, there was still negative integration, but much less so. However, the IECs had to subsequently be adjusted because, as discussed earlier, when background correction points are made in regions associated



with IECs, changing the background correction also requires adjusting any associated IECs.

What must be available for your auditor?

Your auditor will ask you the following during an on-site evaluation:

- What approach do you use for background correction (OPBG-1 pt, OPBG- 2 pt , some form of “fitted” correction)?
- For OPBC, can you show me your correction points for _____ (e.g., As, Cr,Ti, Tl)?
- When were these points determined; can you show me documentation?
- Can you explain how these points were selected?
- Can you demonstrate that the locations selected for OPBC are free from interference? (and if not, do you have an IEC in place?)
- Successfully responding to these questions does not necessarily require a lab to analyze any standard mixes, or individual standards, although analysis of high level mixed standards can certainly help to highlight problems with background correction.

Ultimately, you simply need to be able to demonstrate that background correction is reasonably based and not simply, “That’s what the vendor set up”. It is possible, of course, to overcome poorly placed background correction points by creating an IEC to adjust for the under- or over-compensated background. This will be discussed further in a separate article.

Figure 7 is a screen capture of Thermo Scientific’s [ITEVA software](#) for ICP-OES. Similar software features are available with nearly every ICP instrument purchased over the last 10 years or so. Unfortunately much older instruments and some sequential units may not have this capability.

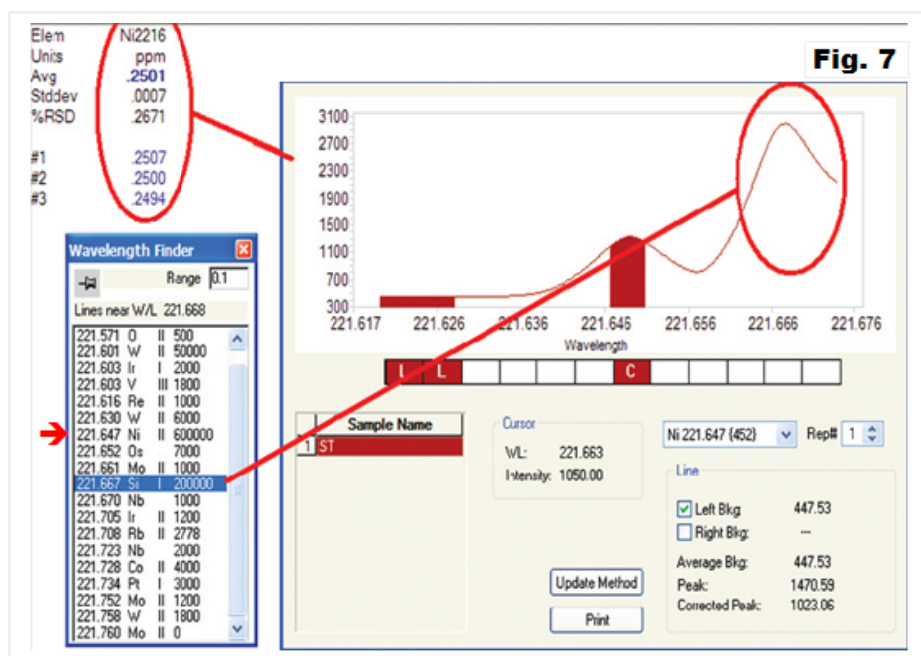
The Thermo Scientific software’s “Wavelength Finder” program, for a given wavelength (here, Ni 221.647), shows nearby emission lines from other elements. In this example, the sample contains not only Ni, but Si, with a wavelength on the right at 221.667 nm. Had

the method included background correction points on either side of the Ni peak maximum, it would be easy for the analyst to remove the right background correction point that would be affected by the Si interference.

Summary

This all explains why your auditor may seem overly concerned with background correction. Your report will likely indicate that, **ICP background correction points have not been properly established or documented.** [EPA 200.7 (4.1.4), EPA 6010D (4.1.1.2)]. Labs must be able to retrieve their background correction scheme and demonstrate that the point(s) selected (or algorithm employed) are appropriate.

Sadly, this detail of ICP-OES is quickly becoming a lost art. Like the old telephone game, training has simply not passed on all the salient information. If we are ever to truly have a single nationwide accreditation standard, all auditors must be looking at the same critical things that affect data quality. As the saying goes, the devil is most certainly in the detail.



Corrective Action: How to comply with NR 149 and prevent reoccurrences

... Take steps to avoid systematic failures by taking the time to consider the cause and make necessary changes. Deal with that wasp nest early to avoid getting stung later.



A new edition of LabNotes marks a convenient time to include an article on corrective action since it is common for us auditors to see unresolved problems and insufficient corrective actions taken and documented, when we visit laboratories. We have noticed that the same data qualifiers are repeatedly associated with reported sample results. Through our lab to lab travels, we have seen systems in place that work really well, and this is our opportunity to share these ideas, along with reminders of the basic requirements.

In NR 149, our administrative rule, corrective action requirements can be found in section **NR 149.38**, sections 1-4. The sections are summarized as follows:

1. Take corrective action for departures from procedures and when QC samples fail to meet limits.
2. Identify the source of the problem, correct the problem; and have a system in place for reviewing that the corrective action taken had the desired effect (the cause of the problem is not reoccurring).
3. Document the corrective actions taken (*the corrective actions/changes must be done in an expeditious manner* - before affected results are released or reported);
4. Monitor the effectiveness of the changes; take additional corrective action if the change did not resolve the problem.

Note: On the corrective action logbook sheet that we provide to labs there are headers in each column that ask specifically 'Did the fix work?' and 'How do you know the fix worked?' to make sure there is a reminder to assess those elements and document them.

Often this seems easier said than done – but **do not fall into the trap of excuses!**

Unfortunately we have noticed that many labs have a long list of non-conformances (failures or departures) that occur - but rarely do we see any real corrective action or change taken, even if they are repeating failures.

You are NOT meeting NR 149 requirements IF... the *only* time a corrective action is documented is when a client complains or a PT fails or in response to an evaluation finding.

Whether it is a matter of:

- Not knowing the approach to take
- The quantity / complexity of the failures are overwhelming
- A hasty decision that the failure does not require corrective action/change (or that it be documented)
- Assuming the problem will go away on its own

These can be handled *if the time* it takes to do it *is taken*. If failures are not ignored (or even better, headed off at the pass - by preventative action), then there will be fewer problems. Fewer problems leads to reduced time spent qualifying data (and calls from data users), less documentation of problems going forward, and less time explaining why these problems have not been fixed to your auditor. An active QA program is **vital** to the success of a laboratory.

Make sure those on the front line (technicians and analysts) are strongly encouraged to take the time to **study the cause of the problem** and **propose the changes** that will eliminate or reduce the likelihood of re-occurrence. As Jon Stewart says, "*If you smell something, say something*".



Larger commercial labs that do a lot of analysis may be using their LIMS to generate summary information that the Quality Manager can use as a review to spot *trends* in the data.

An auditor recently visited a lab that had a very *good* system in place to quickly identify problems (and potential problems). The Quality Assurance Department ran a weekly report from their LIMS, and this report presented for each parameter whenever: the control limit was exceeded once,

or when the *warning limits* were exceeded 4 times in a row. This report is provided back to the lab departments and corrective action was required.


A note on *warning limits*- *warning limits* are not as wide as the control limits. This is well explained in many sources, one of which is Standard Methods. It's as simple as it's named, it's meant to be a 'warning'.

This approach is great because it is not only requiring corrective actions when there is a QC failure, it goes one step further since they are also watching for trends by looking at the repeated warning limit exceedances that appear to be leading to another failure.

one-time occurrence, and chronic would be ongoing (repeating) failures that point to a systemic issue. If a data reviewer notices that one out of every four times a standard is failing low, they may question why this chronic problem has not been worked on and corrected.

- ★ Labs must document the acute failures, since they could be the beginning indicator of a bigger (or chronic) problem.

We have a few examples of corrective actions below – we also appreciate hearing from labs when solutions have been found to quality control problems.



Typical failures that require corrective action:

- Calibration failures
- Laboratory control sample failures
- Method blanks failures (and the sample results are affected)
- Surrogate or internal standards failures
- % RPD limit failures (also applies to column confirmations)
- Interference control sample failures
- Samples and QC that are not prepared, analyzed and evaluated per the method

A couple of comments:

- ★ When corrective actions taken are incomplete (not getting to the real cause), it's a little bit like continuously swatting at a few wasps instead of properly dealing with the nest. For example, a lab that has a problem with poor BOD blanks, and never solves the problem of calibration accuracy, is producing poor BOD results.
- ★ Make sure lab staff understand that corrective action is about having them help supervisors and quality managers get the information they need.
- ★ While a failure requires qualification, this does not eliminate the need for corrective action.
- ★ Corrective actions are not limited to QC limits (e.g. when the SOP includes info that is not correct).
- ★ Sometimes we use terms like acute and chronic (kind of like how a doctor may describe pain...). Acute would be a

Corrective action example 1

" Sometimes you could tell what it was about ... and sometimes it was quite obvious that someone had lost it and it was on an endless loop."

- Todd Rundgren

A. What was the problem?

BOD probe calibration problems were not resolved (an audit finding). This was indicated by erratic blank results which were frequently substantially either more negative or positive than 0.4 mg/L. This was the second repeat finding and the data generated for over three years has likely been affected. Since the blanks are highly negative and erratic, the first issue to resolve was the calibration.

B. What caused the problem?

There are five operators, two DO probes/meters and BOD is analyzed on multiple shifts. It is difficult to have consistency in the calibration with these variables that are not controlled.

C. What was done to try and fix the problem?

The audit finding indicated the importance of consistency with calibration, which could be done by making sure a procedure, placed within easy access, was followed **exactly** by all staff. In addition, all staff attended a meeting to discuss the importance of a good DO probe calibration.

These 10 steps were clearly written and accessible for a water saturated air calibration:

1. Fill a bottle with DI water to the line indicated (1" from the bottom).
2. Cap and vigorously shake the bottle.
3. Take a Kimwipe and dab (very gently, not rubbing the membrane) any droplet of water from the probe.
4. Place the probe in the bottle quickly.
5. Let the bottle sit for exactly 30 minutes using the egg timer on the bench.
6. Make sure the room temperature meets the requirements of 17-23°C and record.
7. Record barometric pressure from the meter.
8. Press the Calibrate button and record.
9. Compare the calibration value to the oxygen saturation chart using the barometric pressure and DO meter calibration temperature. If the calibration value is off by more than 0.2, check for problems.
10. If the problem can't be solved let the supervisor know.

D. Monitor the Corrective Action

Each day's calibration is reviewed to make sure it is within expectations compared to the oxygen saturation point. There was only one time in 20 sets that the blank was just high, 0.3 mg/L.

The calibrations have been determined to be done well and in control, the problem has been fixed as seen by the stable blank results (and lack of negative blanks). *Eventually the lab went the next step when replacing equipment by purchasing an LDO probe.*

Did it fix it?
How do you know?

Corrective action example 2

"If you do not know how to ask the right question, you discover nothing."

– W. Edwards Deming.

A. What was the problem?

The ICP LCS failed for beryllium (Be), at 80% recovery. The control limits are 85-115%.

B. What caused the problem?

Not sure why Be failed, all other parameters recovered well (~95-105%). The other standard solutions passed. Re -prepared and reanalyzed the samples for Be. The next LCS passed.

...This is an example where the cause isn't really dealt with and it is chalked up to a random occurrence. The analyst needs to ask more questions...experience helps here too, if all the other parameters passed, it's even more interesting of a problem that only Be failed. One key to ICP analysis is interferences – method 200.7 lists V and Ce as interferences, but looking at 6010B, titanium is listed as an interference.

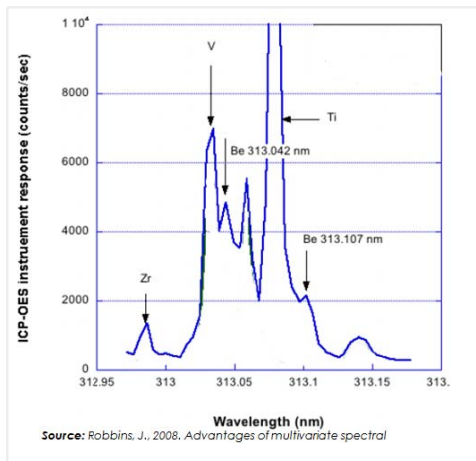
Taking another swipe at this...

Reviewed the peak for beryllium in the LCS. There is a titanium (Ti) peak to the left of Be 313.107 (see image below). The LCS that failed contained both Ti and Be mixed in the same solution (which is a different mix from the calibration and verification standards). Typically titanium is not included in the LCS with Be since it is rarely requested for analysis. But still ...how did this not show up in the interference studies?

Reviewed the interference study data, there was no data that showed there was an interference for Be from Ti! Reviewing spectra from the interference study there was one background point on the right side of Be peak, and **now there are two background correction points, one added to the left**. Adding the background correction point to the left caused the problem when there is Ti present, since that intensity is subtracted out ...resulting in a low result.

C. What was done to try and fix the problem?

Removed the left side background correction point. Analyzed a Ti standard at 50 ppm and there was no interference on Be 313.107. Reanalyzed the LCS that had previously failed low, now the recovery of all parameters pass 85-115% and Be recovery was 92%. Removing the added correction point fixed the problem, which is seen with the passing LCS.



D. Monitor the Corrective Action (did the change fix the problem and how do you know it fixed the problem)?

After 10 analysis sets of an LCS that includes Ti, there have been no failures for Be.

Note: This problem is also about making sure that the instrument method settings are not changed without making sure all the basics for ensuring quality ICF analysis can be met (along with training the analyst on the pitfalls of background correction points along with why certain parameters when in the same solution can cause a problem). Get to the root of the problem (and stop swatting at the wasps). If there is a mental block when trying to resolve a problem, it's always good to go back to the basics of the test and review the method along with contacting the instrument manufacturer. If this does not help resolve the problem feel free to call or email one of the lab certification program contacts.

Make sure to visit our website:
<http://dnr.wi.gov/regulations/labcert/Resources.html>
 There is valuable information in guidance and training that discusses real issues that can occur in any lab (also see the detailed information on ICP).

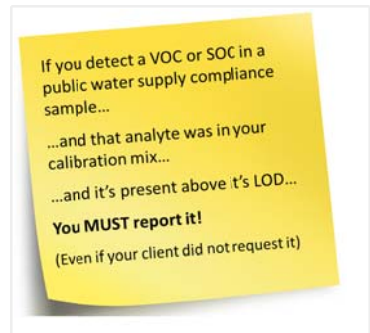
Reporting results for non-“target” analyte detects in Public Water Supply samples

The Bottom Line

- When a compliance drinking water (public water supply) sample is analyzed for SOCs (synthetic organic contaminant) or VOCs (volatile organic contaminant) the laboratory must report any compound detected, at or

above the analyte LOD, for which the instrument was calibrated – *regardless* of the compounds requested to be reported by the client.

- This is a requirement specified under NR 809, Safe Drinking Water, Wisconsin Administrative Code, the state implementation rule of the federal Safe Drinking Water Act. If the calibration and other QC for the non-requested compounds are in control - the result is reported as a quantitative result, and it is reported with the appropriate data qualifier.
- If the calibration and other QC for the non-requested compounds are **NOT** in control, the result is reported as an estimated result, and is then reported with the data qualifier '0' indicating that the result was either Non-numeric or Not Verified.



Pertinent Administrative Rule (Ch. NR 809)

NR 809.207 Compliance requirements for synthetic organic contaminants

(2) DETECTION OF SYNTHETIC CONTAMINANTS NOT LISTED IN S.NR 809.20 (1).

Any detection of a synthetic organic contaminant not listed in s. NR 809.20 (1) shall be reported to the department with the other monitoring reports required under this section. The laboratory shall indicate whether any detected synthetic organic contaminant not listed in s. NR 809.20 (1) has been confirmed or tentatively identified, and when a numerical result is reported, whether the result is quantitative or an estimate.

NR 809.247 Compliance requirements for volatile organic contaminants

(2) DETECTION OF VOLATILE CONTAMINANTS NOT LISTED IN S. NR 809.24.

Any detection of a volatile organic contaminant not listed in s. NR 809.24 shall be reported to the department with the other monitoring reports required under this section. The laboratory shall indicate whether any detected volatile organic has been confirmed or tentatively identified, and

when a numerical result is reported, whether the result is quantitative or an estimate.

What about TICs?

Tentatively Identified Compounds (TICs) that can be determined when using GCMS instruments are not required to be reported. However, we recommend that if a laboratory does detect a TIC at a high concentration that it would report the TIC result - as it may have an impact on public health.

Fine. But how do we report these extra analytes electronically?

All public water supply drinking water compliance results must be submitted to the WDNR electronically through the LDES system. There are fields available for all drinking water compounds to be reported along with a comment field for data qualifiers.

The instruction for reporting these extra compounds are as follows:

39175	VINYLCHLORIDE			0.2	UG/L	v	
79724	XYLENE TOTAL			10000	UG/L	v	
<input type="button" value="Save Sample Without Submitting"/> <input type="button" value="Save and Submit"/> <input type="button" value="Add a Result"/> <input type="button" value="Discard This Sample"/>							

[Sample List](#)

At the bottom of the list of results, click on the "Add a Result" button.

A screen appears giving you two options: "Select Storet Code to Add" or "Find Stores":

Select Storet Code to Add

Enter Storet Code to Add:

Find Stores

Enter Part of Storet description, then click Find:

If you know the Storet code, you can enter that in the "Enter Storet code to add:" field and click on the "Add this Storet" button. For example, if you wish to add a result for Tert-butylbenzene and know the Storet code, enter it and click the "Add this Storet" button, like this:

Select Storet Code to Add

Enter Storet Code to Add:

If you don't know the Storet code, enter part of the description in the "Enter part of Storet description, then click find:" field and click the "Find" button. A list of parameters that match the description you entered will be returned. Click the "select" button for the parameter you wish to report. For example, if you wish to add a result for Tert-butylbenzene, enter a portion of the name, click "Find", and then select the parameter from the list that appears, like this:

Find Storets

Enter part of Storet description, then click find:

Select	Storet Code	Parameter
<input type="button" value="Select"/>	77342	N-BUTYLBENZENE
<input type="button" value="Select"/>	77353	BUTYLBENZENE TERT
<input type="button" value="Select"/>	77350	BUTYLBENZENE SEC

After either option, you will be returned to the result page and the parameter you added will be at the bottom of the list. You can continue adding the result as you did for the others.

39175	VINYLCHLORIDE			0.2	UG/L	v	
79724	XYLENE TOTAL			10000	UG/L	v	
77353	BUTYLBENZENE TERT						
<input type="button" value="Save Sample Without Submitting"/> <input type="button" value="Save and Submit"/> <input type="button" value="Add a Result"/> <input type="button" value="Discard This Sample"/>							

[Sample List](#)

Note: if the calibration and other QC for the non-requested compounds are **NOT** in control, the result is reported with "0", meaning "Non-numeric or Not Verified", as the data qualifier. If the compounds **ARE** in control, use the appropriate data qualifier as you would for any of the requested compounds. The text box shown below is displayed as a dropdown list identified in

0, Non-Numeric or Not Verified
 1, Normal (No problem with sample)
 2, Non-Detect
 3, Between LOD & LOQ
 5, Too much contaminant to quatify
 6, Screen Detect
 F, Field Result

the yellow field column in the screenshot immediately above.

The impact

This article is presented to make sure all drinking water laboratories are aware of these public water supply drinking water compliance

requirements. If not, this is the opportunity for the laboratory to become compliant - if it was unaware of these requirements. These requirements will be assessed during future on-site evaluations and can result in a deficiency if compliance mechanisms are not in place to address these requirements.

If you have any questions at all on this requirement please contact someone in the Laboratory Certification and Registration Program.

2016 Environmental Fee Invoices

... Don't forget to include the payment stub!

May is less than six months away! It's never too early to start prepping folks for the annual spring environmental fees, especially when the process changes and requires recipients to take specific actions to ensure their payment gets credited properly.

The State of Wisconsin has implemented a new financial system to process all invoices. For your payment to be processed on time and to avoid a possible late fee you **must include the payment stub** at the bottom of your invoice.

To save time you can pay your invoice online by going to <http://dnr.wi.gov/epay/>. *Note that the invoice number (your facility's 9 digit FID plus "-2016") and the payment amount must be exactly as it appears on your payment stub.*

Along with these changes the DNR Environmental Fees Program is trying to go green by emailing your invoices.

To ensure quality on time service please assist with the following:


- ✓ **Ensure that we have the most current email address on file**
- ✓ **If your email address changes before May 2016 please inform us of these changes**
- ✓ **Please add DNREnvironmentalFees@wisconsin.gov to your contacts list and mark as not SPAM**

If you have any questions or concerns please contact Jeremy Kahl at (608) 261-4922 or by email at Jeremy.Kahl@wisconsin.gov

Detach and enclose this portion with your check payable to Wisconsin DNR

YOUR NAME	111222330-2016
ACME LABORATORIES	5/27/16
ANY STREET	1,113.50
BOX XYZ	6/30/16
YOUR CITY, WI 55555	

Please mail to:
Wisconsin DNR - Environmental Fees
Drawer #192
Milwaukee, WI 53293-0192



Selected ion monitoring (SIM) GC/MS?

... You gotta tuna this fish.



The use of GC/MS SIM mode is becoming more and more prevalent. As labs struggle to generate lower LODs required to meet action limits, GC/MS SIM has become the preferred option to using conventional detector GC methods. Some of the more common applications for which we encounter data generated using SIM mode are:

- Polynuclear aromatic hydrocarbons (PAHs),
- Pentachlorophenol, and
- 1,4-dioxane

Although it may seem counter-intuitive to incorporate a full scan mode tune before switching over to SIM mode for SIM analysis, it **is** required. Though that could change somewhere down the road, until there is national consensus, approved and promulgated by the EPA, that is the unfortunate reality.

Before the arguments begin, let's back up a bit. To make sense of this, we need to make a quick trip in the [WayBack Machine](#). We need to revisit the whole



concept of tuning. Understanding why we do what we do now (e.g., BFB, DFTPP) will help us better understand where we need to be with respect to tuning prior to performing GC/MS SIM.

A GC/MS system is initially "auto-tuned" using PFTBA (perfloro-tri-nbutyl amine) as a means of optimizing the instrument for maximum sensitivity while providing mass resolution for specific masses designated in the tune algorithm over the range of masses being analyzed. Once tuned, the analyst then runs a specific analytical method.

Two main GC/MS analytical methods were ultimately developed, one for volatiles and another for semivolatiles. Both of these methods were developed as "full scan" GC/MS. Although SIM has been around for 15 years or so, it is just now becoming widely used. Back in the late 1980's, as part of the CLP (Superfund) program, the EPA established "target" tune criteria for BFB (volatiles) and DFTPP (semivolatiles) as a mechanism to ensure generation of uniform mass spectra among all laboratories generating and reporting environmental sample results for compliance testing. These **target tunes** were designed to **actually de-tune the mass spectrometer** to meet specific criteria established for the fragmentation pattern of either BFB or DFTPP.

It's this de-tuning process that sparks discussions when we audit labs performing SIM analysis. We get it. Sensitivity is critical for all target masses (in order to obtain the desired lower LODs), and thus "de-tuning" the instrument is counter-productive. Additionally, with SIM we are focusing on a handful of key masses (actually, m/z) and so other interferent or non-essential masses are filtered out. These are the arguments used to support a position that conventional BFB/DFTPP target tunes have no relevance to SIM applications.

We agree that using conventional BFB/DFTPP tuning criteria is inherently problematic, but we lack an acknowledged alternate approach. We're hoping that we can all agree that some tuning process is required.

We posed the question to the EPA's Methods Information Communication Exchange (MICE), although it was framed in terms of method 8270, as that is the base method typically used for SIM. MICE provided the following response (which we modified to include 8260) – with which we agree and we will hold labs to.

Methods 8260 and 8270 require a tune check be performed prior to analysis, note the use of the word "must" below:

- *...GC/MS system **must** be hardware-tuned to meet the criteria ... for a 50 ng injection of BFB/DFTPP. Analyses must not begin until the tuning criteria are met. [8260B § 7.3.1, 8270C § 7.3.1]*
- *The GC/MS **must** be tuned to meet the recommended BFB [DFTPP] criteria prior to the initial calibration and for each 12-hr period during which analyses are performed. [8260C § 9.2, 8270D § 9.3]*

MICE went on to add that,

Because the word "must" is used in the guidance and not "should" or "may", a tune has to be performed by full scan, even for SIM work. *The tune requirement is not just for library checks, but is also designed to demonstrate that the mass spectrometer is in control. Many laboratories have suggested that documenting a successful auto-tune report at the beginning of each shift should work, however the use of phrases such as "must" or "shall" in the methods make the specific procedure unalterable.*

In summary, if you do not perform and pass a DFTPP check, you cannot call the method 8270C or 8270D. It would be considered a modification of the method.

There's no ambivalence in the method language, and, as MICE points out, SW-846 with all its "guidance" disclaimers, states that, "*The words "shall," "must," or "require" are used to indicate **aspects of the method that are considered essential** to its performance, based on sound analytical practices (e.g., an instrument must be calibrated before use).*" Consequently, a tune which meets BFB/DFTPP criteria becomes a requirement of the method, whether used to perform full scan or SIM analysis. **If a laboratory is not performing a full scan mode tune check before each SIM analysis run, it will result in a deficiency.**

The rules may not always make sense, but we do not make the rules; as a primacy state our role is

to enforce the methods consistently. This is where the system breaks down. Some labs feel no tune is necessary, others feel that an autotune is sufficient, and still others suggest some variation in between. How do we establish defensibility or validity of PAH results by SIM analyzed by four different labs with four different approaches to tuning? Consider five sets of PAH SIM data from five different labs:



- Lab A chooses not to tune at all.
- Lab B opts to perform an auto-tune using PFTBA.
- Lab C tunes in full scan mode to meet standard DFPPP criteria.
- Lab D has developed its own tuning criteria focusing on 8-10 m/z ratios ranging from 128 to 228.
- Lab E has developed its own tuning criteria focusing similar to lab D, but has established much broader acceptance criteria.

Are the data from all five labs comparable? Defensible? Which data should be considered acceptable? Being too flexible can be just as damaging as being too rigid. Chaos is not a viable solution to the problem at hand. Clearly some sort of tuning is in order; not tuning at all is simply not an option for defensible data. So, until such time as the EPA develops a generally recognized tune protocol when performing SIM analysis, we are stuck with requiring the tuning procedure associated with the base method (e.g., 8270).

ICP Endgame –Your Interference Check Standards (ICS)

OK...so we now understand that analyzing an "ICS-AB" standard is of no value in demonstrating that you have properly identified and neutralized spectral interferences. So what DO you use? Wait, let's back up just a minute. We first need to BRIEFLY discuss the elephant in the room – interelement correction factors (IEC).



Let's be clear: we are not saying that IECs are required. In fact we have labs that analyze nothing but processed drinking water that are virtually interference free. If you have no interferences at the analyte levels you see in samples, then no interference correction is required. BUT...you DO have to prove that by analyzing *something* (let's call it an interference check standard, or ICS) to demonstrate that at the levels you encounter, you do not experience spectral interference. And let's just cut to the chase, analyzing a single standard containing all potential interferences is simply not an option. Why? Because many (if not most) of the elements interfere with one another. So jumbling all elements into one standard, while expedient, only serves to further muddle the spectral interference picture.

So, the common denominator here is that whether you use an algorithm (e.g., FACT MSF), IECs, or nothing at all to deal with spectral interferences, you must still provide data to demonstrate that your data is not biased due to spectral interference.

So...what do we require? How does one devise an ironclad collection of ICS "cocktails" that will ensure that your data can stand up to scrutiny regarding spectral interference related bias? First, there is no single solution that fits all. That's the major flaw in the EPA's Contract Lab Program (CLP) [ICSA, ICS-AB] approach. It depends on the resolution of your instrument, the wavelengths you choose, and your background correction protocol. While element "X" may suffer from interference from element "Y" at wavelength "1", switching to wavelength "2" may prove interference free from element "Y", but suddenly element "Z" may become an interferent.

Building the perfect ICS system

1. One size does not fit all.

As discussed above, creating a chef's salad of all the possible interferences in one mix is simply not a viable solution. This is precisely why both methods 200.7 and 6010 spell out a list of 5 or 6 (6010) calibration mixes.



200.7 TABLE 3: MIXED STANDARD SOLUTIONS

Solution	Analytes
I	Ag, As, B, Ba, Ca, Cd, Cu, Mn, Sb, and Se
II	K, Li, Mo, Na, Sr, and Ti
III	Co, P, V, and Ce
IV	Al, Cr, Hg, SiO ₂ , Sn, and Zn
V	Be, Fe, Mg, Ni, Pb, and Tl

6010B TABLE 3
MIXED STANDARD SOLUTIONS

Solution	Elements
I	Be, Cd, Mn, Pb, Se and Zn
II	Ba, Co, Cu, Fe, and V
III	As, Mo
IV	Al, Ca, Cr, K, Na, Ni, Li, and Sr
V	Ag (see "NOTE" to Section 5.4), Mg, Sb, and Tl
VI	P

If verifying lack of interference is difficult when analyzing a single standard containing all elements, how could that possibly work for calibration? It doesn't. And the two methods suggest different groupings of standards because each of the methods recommends different analytes and wavelengths.

2. Defensibility extends only to the concentration levels tested.

This one should be self-explanatory, but that has not proved to be the case. If one of your ICS standards contains element "X" at 10 ppm, that is the limit to which your interference correction for "X" has been verified. Consequently, if—down the road—you analyze a sample which contains 30 ppm (or even 21 ppm) of "X", you no longer have assurance that any correction factors or algorithm associated with "X" hold true at the level identified in the sample. Therefore the appropriate corrective is to immediately analyze a single element standard of "X" at a level higher than that found in the sample to evaluate the potential for over or under-correction.



3. If you don't test it, you cannot prove it doesn't interfere.

The analogy of the age old question, "If a tree falls in the forest...." fits quite well here. A

great example here is Cerium (Ce), a rare earth metal. Cerium interferes with at least 12 common elements using 200.7 wavelengths, yet only rarely have we seen any lab perform interference testing (or verification) for Ce. The reason: "No one ever asks for it and it's rare anyway". Not so true anymore. Ce is being used heavily in phosphorus removal during wastewater treatment. Therefore biosolids will increasingly contain high levels of Ce, which, if uncorrected, will interfere with many target analytes, resulting in biased biosolids metals data.

In recent training on ICP conducted by our program, a challenge PT sample was developed and incorporated into the session. Nine of the 12 labs who reported Vanadium (V) results on this "challenge" PT sample failed because of inappropriately placed background correction points or lack of spectral correction from Molybdenum (Mo) interference. We found that a number of labs had not even incorporated Mo, despite the fact that it is clearly listed as an interference at the main wavelength identified in method 200.7. They didn't worry about it because they "typically don't encounter high levels of Mo". The simple reality is that even the more "rare" elements are becoming more and more common in the environment.

Your auditor is going to ask for your most recent single element interference study and then the associated LOQs for those elements and review them to see what elements demonstrate significant interferences. If they are significant, they need to be tested using an ICS.

4. Interferents should be recovered as well as in an ICV.

The old CLP protocol for evaluation of ICS-A and (*gulp!*) ICS-AB is $\pm 20\%$ of true value. Not good enough. ICS standards are really no different than an initial calibration verification (ICV) standard...right? You spike an ICS-A standard with Al, Ca and Mg at 500 ppm and Fe at 200 ppm and then $\pm 20\%$ is good enough? An Al standard at 500 ppm would mean acceptance criteria of ± 100 ppm! Really? Isn't ICP technology more accurate than that? That's broader criteria than any PT sample! At those levels, we should expect $\pm 5\%$...or 475 to 525 ppm for a 500 ppm

standard. That's not only reasonable, it's appropriate.

So for interferences spiked into each ICS, recovery should be $\pm 5\%$.

5. What's NOT present is more important than what is.

The flaw of the CLP protocol for evaluating interference check standards is that it focuses on recoveries of spiked analytes rather than how spiked analytes impact elements that should NOT be present in the sample. If you are trying to verify that your correction for Mo on V is adequate, wouldn't you want V to be absent from the sample? Then any V that is detected should be attributable to interference that has not been properly corrected. The concentration of any analyte which is NOT present in an ICS should be the same as the concentration of that analyte in an initial calibration blank (ICB). And keep in mind that overly negative concentrations are just as telling as overly positive ones.

6. Too many cooks spoil the broth and too many interferences produce chaos.

Everyone wants to minimize the analysis of standards and QC samples (i.e., "non-billable" samples)...right? That's why instead of multiple standard mixes, we see labs trying to cram everything into one sample. Sure...it eases your workload from the number of analyses standpoint, but it only increases your workload from a data quality standpoint. This is particularly the case when labs try to jumble all the interferent analytes into a single ICS solution.



Consider an ICS containing just Cr, Fe, Mo, and Ti. Boron (B) is one of the unspiked analytes, yet it is detected well above its LOQ. So the alarm bells go off and you determine that correction for interferences on B is inadequate. That's great, but it's only half the battle. WHICH interferent correction is problematic?

The problem here is that B has two prominent lines at 249.677 and 249.772. The 249.677 line

has interference from Mo and Cr within 0.05 nm below the peak and from Ti and Fe within 0.01-0.1 nm above. While 0.1 nm may seem safe, it may not be if the Fe concentration is particularly high. The other wavelength for B suffers similar interference from Ti, Fe, and Mo. You can always use another wavelength...right? Maybe. Does that wavelength provide adequate LOD? And more importantly, what potential interferences are there on **that** wavelength? Yes, it's complex, and devising a quality scheme for evaluating interference correction can be challenging. But who said ICP analysis was easy?

7. Acceptance criteria cannot exceed the LOQ for unspiked analytes.

While in theory establishing criteria for unspiked analytes in an ICS should be \pm LOD, in practice that may not be realistic. But certainly, any unspiked analyte in an ICS should not be outside of \pm LOQ. From that point, the onus is on the lab to determine whether its LOD (and thus LOQ) is unrealistic or if there is a problem with background correction and/or interference correction for the affected analyte.

8. The special case: My samples have no interferences.

There are some labs whose samples are legitimately interference free. An example might be a lab that solely analyzes finished drinking water from a public water supply. There might be naturally occurring Ca and Mg as well as bicarbonate and carbonate. If the water has been softened, there may be minimal Ca and Mg, but a prevalence of Na instead. But ultimately, these samples would be expected to be interference free...right? So what does a lab do if they have established that correction factors are not needed?

Both 200.7 and 6010D (and 6010D is pretty unequivocal) indicate that even if no interference correction is used, the lab still has a responsibility to "prove" that none is needed. In this case an appropriate ICS solution may be one that contains the common cations they find at the highest levels they typically see.

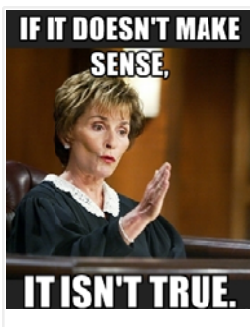
9. **Do it daily for best results.**

Run your series of ICS solutions (because by now you see that one ICS solution just will not cut it for 99% of labs) daily for best effect.



Sure, the reference methods have provisions for running these less frequently if you prove repeatedly that everything is in order. But at the end of the day, the validity and defensibility of each's days analyses depend on your ability to document on each day that interferences are under control.

So that's the recipe for cooking up a quality, defensible interference check protocol. We can't be more prescriptive (*and would you really want us to?*) because every lab's instrument, resolution, and wavelengths are different. That's why the concept of purchasing those ready-made ICS-A and—(*nope, not going there!*) solutions just doesn't make sense....and we all know what Judge Judy says about that! Perhaps everyone should analyze an ICS-A, containing Ca, Mg, and Al at about 500 ppm and Fe at about 200 ppm, but there needs to be more. At least an ICS-B and more likely an ICS-C and ICS-D as well.



*****Remember*****

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