September 2013
Revisiting Linearity of Recovery Studies

In the past I have questioned the value of doing sampling recovery studies at multiple levels, as well as doing so to demonstrate linearity (see the Cleaning Memos for July 2007, July 2009, and October 2010). My assumption when talking about linearity was my perception that people were expecting a significantly changed (decreasing or increasing) percent recovery as the spiked level increases. While I believe that might be the case when the different spiked levels are different by factors of 10 or 20, it is generally not the case that people do multiple spiked levels over such a broad range. A more typical procedure is to utilize spiked levels of 50%, 75%, 100%, 125%, and 150% of the target residue limit. In reviewing some of the publications (mentioned in the Cleaning Memo for October 2010), at least some appear to show a clear linear relationship, but that linear relationship is a flat line (slope of "zero"). In other words, that data show that essentially at those slightly different spiked levels, the percent recoveries are essentially the same (within experimental error or variation). So, if what is meant by "linearity" is that recoveries at different spiked levels will be essentially the same, and that the linear result is a straight line with zero slope, I must modify my past assertions that linearity is not an expectation (at least over a relatively narrow range).

However, I still maintain that there is little value in doing recovery studies at multiple spiked levels. If the recoveries are going to be linear, meaning that each will be essentially the same recovery percentage, then just doing one spiked level should be adequate to determine what the "official" percent recovery is for that sampling method and that recovery (as well as for that material of construction).

I also continue to maintain that if recoveries were done at significantly different spiked levels (different by factors of at least 10 or 20), then good science and logic tells us that as the spiked level increases, the percent recovery will decrease (see the Cleaning Memo of July 2007 for rationale for this). It is for this reason that I still believe that the best procedure is to perform recoveries at one level, and that level is preferably at the residue limit. Rather than doing multiple spiked levels, I think that more replicates at one level gives a better picture of the extent of variability of the sampling process.

Furthermore, if the actual sample analytical result is lower than the residue limit by a factor of 10 or more, then the recovery study done at the limit (a higher spiked level) represents a potentially lower percentage, and therefore a worst case. I would prefer not to do a spiked recovery at a level equivalent to 10% of the residue limit. The reason for this is that (other things being equal), I would tend to get more variability in the analytical result at lower values as compared to results at higher levels. Consider the case of Total Organic Carbon (TOC), where my residue limit in the tested sample is 2 ppm (this is just given as an example). Suppose my actual sample in my cleaning process protocol was as expected to be as low as low as 0.2 ppm TOC. If I tried to do spiking levels where the expected 100% recovery result was only 0.2 ppm, I could expect significant variability for TOC at 0.2 ppm (as compared to 2 ppm). And again, the higher spiked level represents a worst case (lower value) for the percent recovery, and therefore can be applied to the lower level.

However, the main point of this Cleaning Memo is to point out my changed view, that linearity of multiple spiked levels is generally reasonable, provided that the expectation is that that linear relationship has a slope of zero (meaning that the percent recoveries are the same over a relatively small spiked range).