

## September 2002 Sampling the Sampling Rinse

I have discussed in most of my cleaning validation training seminars that there are two types of rinse sampling. One type (the more conventional) involves taking a “grab” sample at the end of the process rinse. A second type is a “sampling rinse”, in which a separate, discrete rinse (following the final process rinse) is utilized during the cleaning validation PQ runs. A “sampling rinse” may involve sampling a small part, such as a filling needle, by passing a fixed volume of the sampling liquid (for example, WFI) at a designated temperature through the filling needle, and collecting the sampling liquid in a suitable vessel as it exits the tip of the filling needle. Or, the “sampling rinse” may involve a larger surface, such as a 5000 L manufacturing vessel, which is sampled by utilizing the CIP system to pass a fixed volume of the sampling liquid (for example, WFI) at a designated temperature through the spray device to cover all internal surfaces of the equipment, and collecting that rinse solution in a suitable collection vessel as it exits the drain valve in the manufacturing vessel.

It is the second type of rinse sample that is the subject of this Cleaning Memo. The specific issue to be discussed is how that sampling rinse itself is sampled for analysis by a suitable analytical technique. The example used to illustrate proper sampling of the “sampling” rinse will be the sampling of a 5000 L vessel with a fixed volume (for example 100 L) of WFI as the sampling rinse.

In this example, it is not appropriate to sample the discrete, separate “sampling rinse” the same as you would sample a grab sample at the end of the process rinse. The appropriate way to sample a “sampling rinse” is to collect the “sampling rinse” in its entirety (that is, in this example, the entire 100 L), mix it until it is uniform, and then take a sample from that uniform, mixed solution to analyze by whatever analytical technique is chosen. The rationale is that if you just sample the final 250 mL of the discrete “sampling rinse” as it exits the vessel, you are not necessarily getting a representative sample containing residues that would be removed from the equipment surfaces during the “sampling rinse”. In such a “sampling rinse”, it can perhaps be expected that most of the residue comes off the surfaces during the early to middle parts of the rinse. In order to quantitate the residue, and relate it to potential contamination of the next product, the sampling volume and the surface area sampled have to be taken into account. Unless the sample taken from the sampling rinse is done correctly, this relationship cannot be established.

It bears repeating that this discussion is focused on situations where a separate, discrete “sampling rinse” is performed after the process rinse is completed. It does not apply to those situations in which the rinse sample is a grab sample at the end of the process rinse itself.