

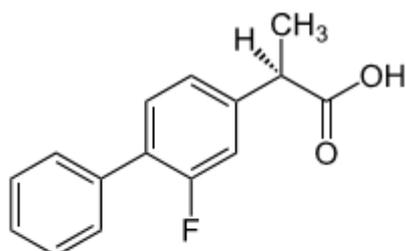
An Improved SPE Clean Up and Recovery of Pharmaceutical Compounds from Plasma using Strata[®] -X

In one corner, we have liquid-liquid extraction (LLE), which is perhaps the most established clean-up technique used in the chromatography field. Although it has been used for years, newer techniques with improved specificity towards particular analytes, have allowed analysts to improve recovery and reproducibility of their samples. So, face your attention to the other corner ladies and gentlemen for solid phase extraction (SPE). With its targeted analyte extraction, this will be one tough match for LLE, but it's time to find out who the champion will be for clean-up and extraction in a pharmaceutical setting!

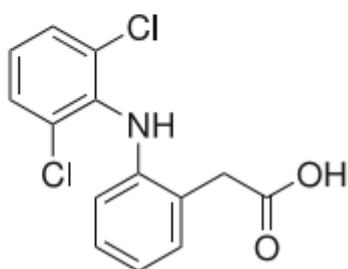
LET'S GET READY TO RUUUMMMBBLLLEEEE!!!!!!!!!!

Round one will consist of the exploration of the two popular extraction methods, SPE and LLE for the isolation of diclofenac from plasma, using a water matrix as the referee (or control). Diclofenac is a slightly acidic non-steroidal anti-inflammatory drug that has been used as a post-operative pain reliever in adult and pediatric patients (**Figure 1**).

Figure 1.
Structure of Internal Standard Flurbiprofen and Diclofenac



Flurbiprofen ($pK_a = 4.2$)



Diclofenac ($pK_a = 4.0$)

We will give them both a fair fight by starting with the same plasma pre-treatment step; filtration through a gauze cloth. Afterwards, 500 μ L of diclofenac, dissolved in 5 % methanol, was added to 500 μ L of plasma, and the solution mixture was then acidified with 600 μ L of 1M phosphoric acid.

Ding-Ding: Round One

SPE steps up first to LLE with pre-treated plasma samples that are further cleaned up and concentrated by a simple Strata-X[®] method consisting of conditioning the cartridge, equilibrating, loading the sample, a washing step, eluting the proper analytes, and a drying down for ultra-concentration.

Oh, but what's this! After pre-treatment, LLE adds 5 mL of hexane IPA (95:5) to the pre-treated solution, which is followed by 1 minute of vortexing, and 10 minutes of centrifugation at 2,000 rpm. The organic layer is then extracted and dried down for

comparable concentration to SPE.

Whistle blows: Round one ends in a tie, both teams fought valiantly and ended up with seemingly clean extracts.

Ding-Ding: Round Two

The veteran in the ring, LLE, steps up with traditional tactics of being used as the standard clean-up procedure for a variety of pharmaceutical samples. Although the technique has been used for many decades, it could stand to be improved upon.

SPE comes in with a right hook and knocks LLE off balance by providing many benefits over LLE's traditional method, including higher recoveries, time and solvent savings, as well as more consistent results.

To kick this match up a notch, recoveries were compared to see which technique, SPE or LLE, reigned supreme. With this added twist, it looks like Strata-X SPE is yielding approximately 86% absolute recovery of 15 µg/mL of diclofenac in the plasma matrix as opposed to 46% for LLE. And the crowd yells "Boo LLE!!!"

This accounts for almost a two-fold increase in recovery as compared to LLE!

Whistle blows: Round two ends with SPE taking the lead!

Figure 2.
Diclofenac Extracted Reference Curve: Solid Phase Extraction in Plasma Matrix

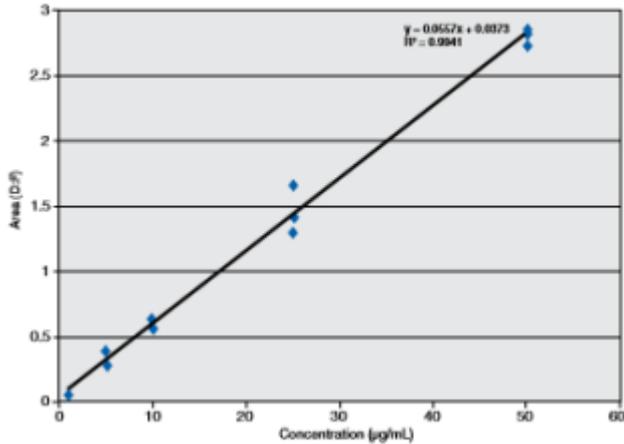
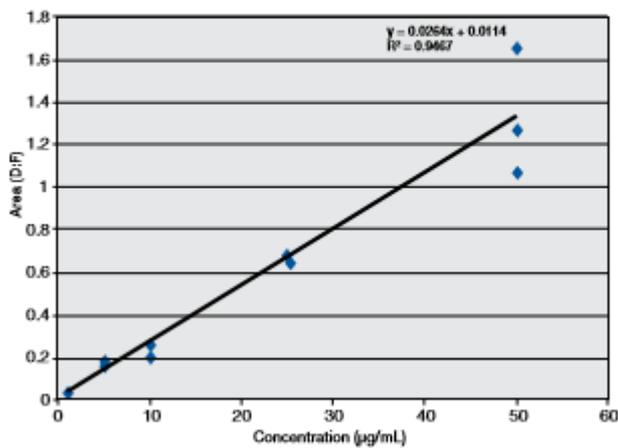


Figure 3.
Diclofenac Extracted Reference Curve: Liquid-Liquid Extraction in Plasma Matrix



SPE provides a greater absolute percent recovery by two-fold over LLE, the Strata-X procedure shows less variability between cartridges. SPE is more precise and reproducible than LLE for the extraction of pharmaceutical compounds.

Ding-Ding: Final Round!

The Strata-X SPE comes in strong, utilizing the chemical bonding properties of a pyrrolidone

ligand, which makes it a prime target for the retention of hydrophobic neutral compounds, while also retaining basic and acidic compounds under strong organic wash conditions.

With the presence of the phenyl ring in the pyrrolidone ligand, LLE doesn't stand a chance against SPE. Pi-pi interactions are prevalent for compounds with aromatic structures, and acidic and basic compounds bind to the nitrogen of the secondary amine and the carbonyl carbon.

LLE comes in with a last-ditch effort punch by utilizing two immiscible solvents that compete for interaction with the analyte of interest.

The punch doesn't land! And with that SPE finishes LLE off and wins the match!

While LLE has been a universal choice of extraction, SPE poses more advantages over LLE for the extraction of diclofenac, via the existence of multiple binding sites on the Strata-X sorbent. This match up shows that SPE provides greater absolute recovery of diclofenac when compared to LLE; and is less time-intensive, consumes less solvent than traditional LLE procedures, and provides better reproducibility, thereby demonstrating that the extraction method of choice for pharmaceuticals, such as diclofenac, is SPE.

Solid Phase Extraction is the *WINNER!!!*



For a more technical look at the comparison of **SPE and LLE methods** in a pharmaceutical setting, see the original application note. If you would like to see how SPE stacks up against your current LLE method request a free sample [here](#).

Share with friends and coworkers:

- [Click to email a link to a friend \(Opens in new window\)](#)
- [Click to share on Twitter \(Opens in new window\)](#)
- [Click to share on Facebook \(Opens in new window\)](#)
- [Click to share on Pinterest \(Opens in new window\)](#)
- [Click to share on LinkedIn \(Opens in new window\)](#)
- [Click to share on Tumblr \(Opens in new window\)](#)
- [Click to share on Reddit \(Opens in new window\)](#)