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OPTIMIZATION OF PARALLEL SOLID PHASE EXTRACTION USING POSITIVE PRESSURE TECHNOLOGY

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LC/MS analysis has become the standard analytical technique for pharmacokinetics replacing HPLC UV based techniques. MS which is a highly selective detection technology allows analytical time to be considerably reduced (from 15 min for HPLC down to 3 min). Parallel sample preparation is necessary to achieve such a high throughput. A standard SPE method with bronchodilators has been set up to demonstrate the suitability of SPE 215 for high throughput sample preparation with various SPE plates (3M Empore• RP C18, Waters OASIS•).

215 SPE with integrated sealing foot design uses the proven positive pressure elution technique to run 8 samples in parallel. Gas is applied on SPE columns after each SPE step to push fluids through the stationary phase. Gas pressure and duration is a crucial parameter to optimize in order to get high extraction recoveries and high reproducibility.

This poster presents the optimization process developed to reach both robust and fast SPE.

20 μg/ml of mixed theobromine, theophylline, hydroxyethyl theophylline and caffeine in reconstructed bovine plasma are extracted using a 5- step SPE process and diluted prior to injection. Air push duration is optimized for each SPE step, graphical data (recoveries and RSD% versus air push duration) will illustrate how this is accomplished. Within-batch recoveries and reproducibility are obtained on the basis of a complete 96 well plate extraction. Recoveries are highly dependent on stationary phase, high reproducibilities (RSD below 5.5% for Empore• 3M plates and below 3% for Waters OASIS• plates) are reached.

Throughput complies with LC/MS analysis but throughput always needs to compromise with recoveries.