Solid Phase Extraction (SPE) Method Development Made Easier

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**Introduction**

Solid phase extraction (SPE) is one of the simplest yet most effective and versatile methods of sample preparation. It is used for both clean-up and pre-concentration of analytes in a variety of matrices. It is simple to perform, allows a single sample to be processed and offers greater flexibility compared to traditional liquid-liquid extraction (LLE). SPE also offers shorter processing times as well as more reproducible results compared to LLE. The use of solid phase extraction has been extensively described in the literature. The method has been applied to a range of analytes and matrices. The solid phase extraction protocol consists of the following steps:

1. **Condition:** 1 ml MeOH.
2. **Load:** 1 ml phosphate saline buffer (pH 7) spiked with 20 – 120 µg/ml std mix.
3. **Wash:** 1 ml Sodium acetate (50 mM, pH 7, 5% MeOH).
4. **Elution:** 20 – 120 µg/ml std mix in saline phosphate buffer spiked at 20 – 120 µg/ml.

**Objectives**

- To evaluate a new ion-exchange solid for the cleanup of neutral and acidic pharmaceutical compounds.
- To evaluate an improved SPE method preparation for the separation of neutral and acidic pharmaceutical compounds.
- To develop a chromatographic system for the separation of neutral and acidic pharmaceutical compounds.
- To optimize the elution conditions.

**Experimental**

- A 300 µl silica-based SPE cartridge was conditioned with 2 ml methanol and 2 ml phosphate saline buffer (pH 7) spiked at 20 – 120 µg/ml.
- The cartridge was then washed with 1 ml of sodium acetate (50 mM, pH 7) and eluted with 1 ml ammonium acetate (50 mM, pH 7) spiked at 20 – 120 µg/ml.

**Results**

- The sorbents were not dried in further experiments.
- Drying gave less than 10% differences in recoveries for neutral compounds.
- Recoveries of acidic compounds reduced by more than 40% after drying.

**Conclusion**

- An SPE method was successfully developed on-column and the optimized conditions transferred to cartridge format.
- Effect of drying the sorbent during the SPE procedure.
- B-Vitamins spiked in saline phosphate buffer spiked at 20 – 120 µg/ml.

**Further work**

- Cleanup of real samples such as biological serum, sewage water etc.

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