

Suggested SPE method for the extraction of Streptomycin from food using SiliCycle® SiliaPrep™ SCX and C18

We describe here a two steps sample preparation method for the determination of “Streptomycin residues in food” using Solid Phase Extraction (SPE) clean-up with Strong Cationic Exchange Cartridge (**SiliaPrep™ SCX**) and Reversed-Phase Cartridge (**SiliaPrep™ C18** (17%)). Analysis of the sample can be done using the analytical technique HPLC (or LC-MS).

This is a recommended sample preparation method, however, this method have not been verified in SiliCycle® laboratories but is based on methods used with similar matrices and analytes. Therefore, before trying the procedure directly on the matrix, you should first develop a procedure using pure solvent containing the analyte. You will find below, tips for the choice of solvent that you should use to establish your SPE method development:

EXTRACTION PROCEDURE

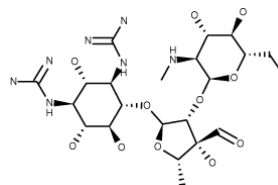
SPE Cartridge types:

- SiliCycle® SiliaPrep™ SCX (500 mg, 6 mL)
PN : SPE-R60530B-06P
- SiliCycle® SiliaPrep™ C18 (17 %) (500 mg, 6 mL)
PN : SPE-R30230B-06P




Note: Other phases can be used for the extraction of this compound (SiliaPrep™ SCX-2 instead of SCX).

Analyte structure:





- Streptomycin residues
[aminoglycoside antibiotic used as a veterinary drug]
























Sample pre-treatment:

- **Sample homogenization is required**
 -  Stir for 5 mins, 5 g of homogenized sample in 20 mL of a solution of HClO₄ (perchloric acid) at pH 2.0 (~0.01 M)
 -  Then centrifuge for 15 mins and filter
 -  Sample ready for the first clean-up

First SPE clean-up using SiliaPrep™ SCX:

- **Column conditioning step**
 -  Solvent : 5 mL of a solution of HClO₄ at pH 2.0
- **Sample loading**
 -  Load directly on the top of the cartridge the sample previously homogenized
- **Interferences washing step**
 -  Solvent : 5 mL of H₂O
- **Streptomycin elution step**
 -  Solvent : 25 mL of a 0.2 M phosphate buffer (pH 8.0) solution

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-  **Streptomycin solution pre-treatment before second clean-up:**
- ③ **Solution pH adjustment required**
 -  Add 2 mL of a 0.5 M solution of AHS (sodium 1-heptane-sulphonate)
 -  Then the pH is adjusted at 2.0 with concentrated H₃PO₄ (phosphoric acid)
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-  **Second SPE clean-up using SiliaPrep™ C18 (17 %):**
- ③ **Column conditioning step**
 -  Solvent 1 : 5 mL of MeOH
 -  Solvent 2 : 10 mL of a solution of 10 mM of AHS (pH 3.3)
 - ③ **Sample loading**
 -  Load directly on the top of the cartridge the previous sample extract
 - ③ **Interferences washing step**
 -  Solvent 1 : 10 mL of H₂O
 -  Solvent 2 : 4 mL TBME(*t*-Butylmethylether)/hexane (4:1 v:v)
 - ③ **Streptomycin elution step**
 -  Solvent : 5 mL of a solution of 10 mM of AHS (in MeOH)
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-  **Analytical method:**
- ③ Generally analysed by HPLC (or LC-MS)
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-  **Reagents used:**
- ③ For this extraction method you will need :
 -  *t*-Butylmethylether (TBME)
 -  Hexane
 -  Methanol
 -  Perchloric acid (HClO₄)
 -  Phosphate buffer solution*
 -  Phosphoric acid (H₃PO₄)
 -  Sodium 1-heptane-sulphonate (AHS)
 -  Water (H₂O)
- *Phosphate buffer solution preparation : dissolve 0.55 g of NaH₂PO₄•H₂O, 2.85 g of Na₂HPO₄•2H₂O and 8.7 g of NaCl in 1L of H₂O.
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-  **General comments:**
- ③ If solvent evaporation is required before the analysis, add 2 mL of a solution of 10 mM AHS at pH 3.3 to avoid the lost of streptomycin due to compound sublimation under vacuum.
 - ③ If the sample matrix is not too complicated, first clean-up using **SiliaPrep™ SCX** can be omitted and the extraction can be done using only the **SiliaPrep™ C18 (17 %)**.
 - ③ SPE volume sizes and sorbent masses can be different; they are related to the sample amount to be purified.
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