

SupelMIP™ SPE – NSAIDs

Product Description:

Molecularly imprinted polymers (MIPs) are a class of highly cross-linked polymer-based molecular recognition elements engineered to bind one target compound or a class of structurally related target compounds with high selectivity. Selectivity is introduced during MIP synthesis in which a template molecule, designed to mimic the analyte, guide the formation of specific cavities or imprints that are sterically and chemically complementary to the target analyte(s). **It is therefore critical for analysts to use the methodology described below when using this phase.** Conventional generic methodologies employed with conventional SPE chemistries (e.g., reversed-phase C18) will yield sub-optimal results when employed with this phase.

The following method(s) have been determined for **non-steroidal anti-inflammatory drugs (NSAIDs)** that can be optimized for a number of matrices. The NSAIDs that we have tested so far include: **Naproxen, Clofibrac Acid, Diclofenac, and Ibuprofen.**

The first procedure is a general procedure that can be followed if a matrix specific method is not included in this data/instruction sheet. This general procedure represents a recommended starting point for further optimization. The general procedure is followed by matrix specific procedures.

For the most recent matrix specific applications, please visit www.sigma-aldrich.com/supelmip and download the most recent version of the data/instruction sheet.

Important Note: The below procedure(s) may require further optimization. A special team of experts in SupelMIP SPE method develop has been formed to offer technical consultation. To reach a SupelMIP technical expert, please visit www.sigma-aldrich.com/supelmip-techsupport and fill out the questionnaire. A SupelMIP scientist will respond within 24 hours (barring holidays).

Protocol for Extraction of NSAIDs – General Procedure:

Sample Pre-treatment

For solid/tissue samples:

1. Homogenize 5.0 g of sample with I.S.; and add 10 mL 10 mM ammonium formate, pH 3
2. Remove particulate via centrifugation or using glass fibre filter (0.7 µm)

For liquid samples:

1. Adjust to pH 3 by 10 mM ammonium formate/formic acid

Note: Deuterated I.S. is recommended for each analyte for accurate quantitation.

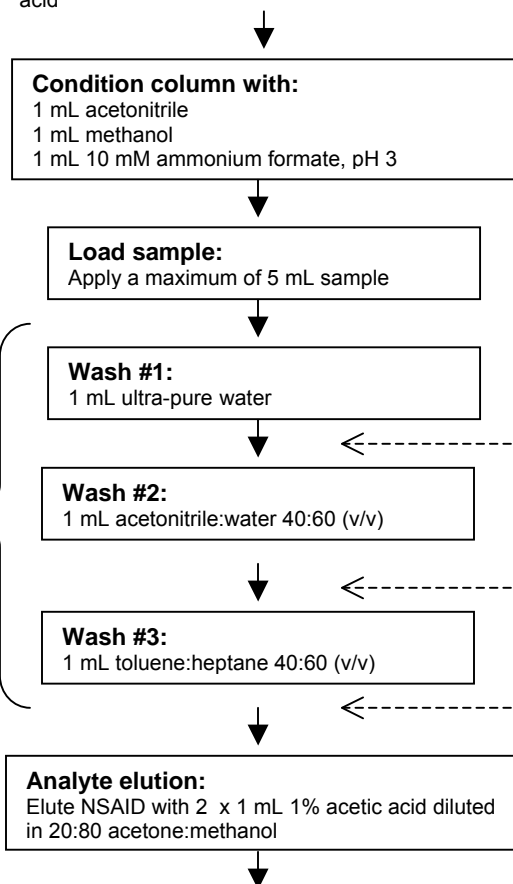
The sample should be completely aqueous prior to SPE processing. No organic modifiers should be present in the sample.

Additional sample pre-treatment may be required depending on the complexity of the sample. For example, a protein ppt step may be necessary for samples that contain high levels of protein.

Recommended flow rate during sample load is ≤ 1 mL/min. If possible use gravity flow during the sample load step.

A flow rate of 0.5-1 mL/min. is recommended for each wash step. The wash steps should be performed in the prescribed order.

Recommended flow rate during elution is ~ 0.2 mL/min



Note: Do not allow the phase to go dry during conditioning. Recondition if the phase goes dry.

Important: Apply a strong vacuum through cartridge for at least **5 min.** to remove residual moisture and ensure a dry cartridge (-0.7 bar, -20 in Hg, or -70 kPa).

Important: Apply a strong vacuum through cartridge for at least **1 min.** to remove residual moisture and ensure a dry cartridge (-0.7 bar, -20 in Hg, or -70 kPa).

Important: Apply a strong vacuum through cartridge for at least **3 min.** to remove residual moisture and ensure a dry cartridge (-0.7 bar, -20 in Hg, or -70 kPa).

Apply a gentle vacuum (-0.4 bar or -12 in Hg for 5-10 s) between each wash step unless noted otherwise.

Gently evaporate the elution solvent to dryness and reconstitute in 150 µL LC-MS mobile phase.

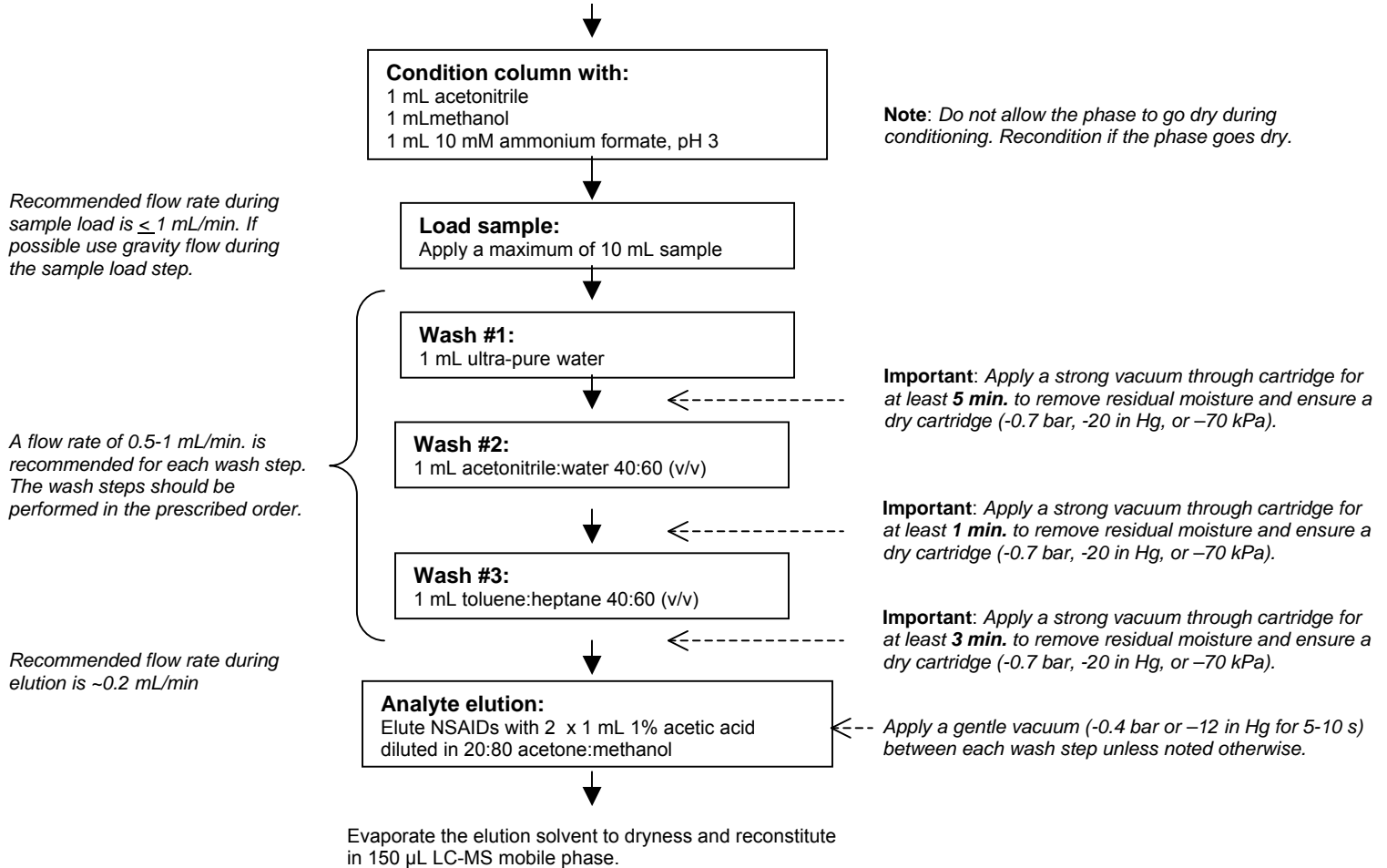
Protocol for Extraction of NSAIDs in Waste Water*:

Sample Pre-treatment

1. Store waste water sample at 4°C under dark conditions prior to analysis.
2. Add I.S. – final concentration 400 ng/mL
3. Filter the sample as necessary using a 0.7 µm glass fiber filter
4. Adjust the sample to pH 3 using 10 mM ammonium formate buffer.

Note: Deuterated I.S. is recommended for each analyte for accurate quantitation.

The sample should be completely aqueous prior to SPE processing. No organic modifiers should be present in the sample.



* Selective determination of acidic pharmaceuticals in wastewater using molecularly imprinted solid-phase extraction. S.Zorita¹, B.Boyd², S.Jönsson², E.Yilmaz², C.Svensson², L.Mathiasson², S.Bergström². 1. Division of Analytical Chemistry, Lund University Lund, Sweden. 2. MIP Technologies AB, Lund, Sweden. Analytica chimica acta 626 (2008) 147–154

Troubleshooting:

Improve Recovery:

- ♦ Do not exceed the recommended load and wash volumes.
- ♦ Minimize flow rate during sample load and elution.
- ♦ Ensure that the sample is pH ≤ 3 prior to sample load.
- ♦ The drying steps described in the wash section are critical for maintaining optimal recovery and selectivity.
- ♦ Do not exceed the acetonitrile level (40:60) described in wash step #2. Higher concentrations of acetonitrile may result in premature analyte elution.
- ♦ Increase elution from 2 x 1 mL to 3 x 1 mL

Recommended Analytical Method:

<p>Recommended Analytical Technique:</p> <p>LC-MS-MS or LC-MS</p>	column:	Ascentis C18, 10 cm x 2.1 mm I.D., 3 µm particle size (581301-U)
	instrument:	LC-MS/MS
	mobile phase:	(A) 5 mM ammonium formate, pH 6 (B) methanol
	gradient:	Min. A% B% 0.0 65 35 13.0 10 90 13.1 65 35 17.0 65 35
	flow rate:	0.2 mL/min.
	temp.:	ambient
	det.:	MS/MS, MRM transitions
		Clofibrac acid 213/127 & 213/85
		Naproxen 229/185 & 229/170
		Diclofenac 294/250 & 294/214
		Ibuprofen 205/161
		Ibuprofen-d3 208/164
	polarity:	Negative
	ion source:	Turbospray
	ion spray voltage:	4000 V
source temp:	500 °C	
nebulizer gas:	60 psi	
curtain gas:	10 psi	
inj.:	30 µL	

Product Information:

Description	Pkg. Qty.	Cat. No.
SupelMIP SPE - NSAIDs		
25 mg/3 mL	50	52769-U
25 mg/10mL (LRC)	50	52772-U
SupelMIP SPE - Nitroimidazoles		
50 mg/3 mL	50	52734-U
SupelMIP SPE - Full Beta-receptors (beta-blockers & beta-agonists)		
25 mg/10 mL (LRC)	50	53223-U
25 mg/3 mL	50	53224-U
SupelMIP SPE - Beta-blocker (class selective)		
25 mg/10 mL (LRC)	50	53218-U
25 mg/3 mL	50	53213-U
SupelMIP SPE - Beta-agonists (class selective)		
25 mg/10 mL (LRC)	50	53202-U
25 mg/3 mL	50	53225-U
SupelMIP SPE - Clenbuterol		
25 mg/10 mL (LRC)	50	53201-U
SupelMIP SPE - TSNAs (NNK, NNN, NAB, NAT)		
50 mg/10 mL (LRC)	50	53221-U
50 mg/3 mL	50	53222-U
SupelMIP SPE – NNAL		
25 mg/10 mL (LRC)	50	53206-U
25 mg/3 mL	50	53203-U
SupelMIP SPE - Chloramphenicol		
25 mg/10 mL (LRC)	50	53210-U
25 mg/3 mL	50	53209-U
SupelMIP SPE – Fluoroquinolones		
25 mg/3 mL	50	53269-U
SupelMIP SPE – Amphetamines (class selective)		
25 mg/3 mL	50	53228-U
SupelMIP SPE - Riboflavin (Vitamin B2)		
25 mg/10 mL (LRC)	50	53207-U
SupelMIP SPE - Triazine 10		
25 mg/10 mL (LRC)	50	53208-U

SupelMIP SPE developed by MIP Technologies AB

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