Extraction of Sodium Monofluoroacetate from Animal Feed

This represents recommendations for SPE method development. The proposed steps are based on experience with similar analytes and matrices, but have not been verified in Argonaut laboratories. Please refer to section below for the analyte and matrix considerations that were made in developing this method.

As for all method development, this procedure should first be developed using pure solvent spiked with analyte. Only after the chemistry is established should spiked matrix samples be tested.

Non-aqueous samples: spike a solvent similar to sample matrix.

Aqueous samples: spike reagent water or 10 to 20 mM buffer. An appropriate buffer is usually the same as that used in the equilibration step.

This method is proposed for the extraction of sodium monofluoroacetate from animal feed using an anion exchange retention mechanism.

**EXTRACTION PROCEDURE**

**ISOLUTE SPE Column**

NH2 (Part # 470-0100-C) and SAX (Part # 500-0100-C) should be tested in parallel.

There may be more than one phase that could be effective in the extraction of this compound. The method development should include testing phases in parallel in order to optimize the procedure.

<table>
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<tr>
<th><strong>Pre-treatment:</strong></th>
<th>Dissolve 0.5 g of ground pellet in 2 ml of 1 mM Tetramethylammonium hydroxide (TMA). Decant liquid. Repeat twice more, with 2 x 2 ml TMA solution. Combine extracts and adjust pH~7 with formic or acetic acid.</th>
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<tr>
<td><strong>Solvation:</strong></td>
<td>Rinse the extraction cartridge with 10 ml of 1 mM (TMA) at 20 ml per minute. Rinse again with 10 ml reagent water at 20 mL per minute. Rinse once more with 10 ml methanol at 20 ml per minute.</td>
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<tr>
<td><strong>Equilibration:</strong></td>
<td>Rinse the extraction cartridge with 10 ml of reagent water at 20 ml per minute to remove excess methanol. Then rinse with 5 to 10 ml of 10 mM formate or acetate buffer, pH~7 at 10 ml per minute to adjust sorbent to sample conditions.</td>
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<tr>
<td><strong>Sample Application:</strong></td>
<td>Sample may be loaded at rates not exceeding 10 ml per minute. After the method chemistry has been optimized, increased loading rates may be tested.</td>
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</table>
Interference elution: Rinse extraction cartridge with 5 ml of 10mM formate or acetate buffer, pH~7 at 10 ml per minute.

Analyte elution: Elute analyte with ethyl acetate, acidified with acetic acid. Elute with 2 x 2 ml of solvent. Dilute to 25 ml or as necessary with ethyl acetate.

Structure

As shown.

Structural considerations

This analyte is quite polar, and is ionizable. It has a pKa of 2.6. At a pH of 5 or greater, it can be extracted via anion exchange.

Matrix considerations

This compound is present in a wholemeal pellet, which can be dissolved into an aqueous medium for extraction. Once the pellet is dissolved, the compound will be in an aqueous matrix, and amenable to ion exchange extraction.

Analytical method

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Reagents

1. 1 mM Tetramethylammonium hydroxide
2. Formic or acetic acid
3. Reagent water
4. Methanol
5. 10 mM formate or acetate buffer, pH~7
6. Ethyl acetate, acidified with acetic acid

General comments

The derivatization and analysis should be carried out as usual.