Sulfate and Phosphate Anion Analysis

MATERIALS

- Pre-Cleaned 4 mL Screw Top Vials, 15 X 45 mm (Sigma, 27340)
- 0.06 N HCl

PROCEDURE

1. Mix sample with an equal volume of 0.06 N HCl and place in a pre-cleaned 4 mL screw top vial, close tightly, and vortex.

2. Place vials in an oven heated to 150 °C for 1 hr.

3. Following hydrolysis allow the sample vial to cool and centrifuge to settle any condensation that may have accumulated on the cap.

4. Bring the samples to dryness either by dry nitrogen flush or using a speed vac.

5. Pyrolyze the dried samples over an open flame for about 15 seconds. Wait until the bottom of the tube turns red hot before starting the countdown.

6. Let the samples stand until it cools to room temperature, then dissolve the dry sample in Milli-Q water. Prepare the appropriate dilutions desired for HPLC injection.

7. Inject into HPLC for analysis

HPLC METHOD

- Column:
  - Dionex IonPac AS11 Analytical Column 4.6 mm x 250 mm, 4μm, with compatible guard column.
- Solvents:
  - A: Water
  - B: 50 mM KOH
  - Initial conditions of 15% B at 1.0 mL/min
- Heat Control:
  - Cell Heater Temperature: 30 °C
  - Temperature Compensation 1.7% per °C
- Suppressed Conductivity Detector Settings:
  - Type: ASRS_4mm
- Current Set: 160 mA
- Suppressor Type: Hydroxide, 40

- Gradient Settings:

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<tr>
<th>Time</th>
<th>%A</th>
<th>%B</th>
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