EPA METHOD 314.0 PERCHLORATE

LAB NAME: _____**DATE:**_____

1. Columns and Reagents Sections 3, 6, 7

- Γ Holding Time 28 days
- Γ Column AS 16
- Γ Volume of sample 1.0 ml
- Γ Eluent – 50 mM sodium hydroxide
- Γ Matrix Pretreatment cartridges – Ba (SO_4), Ag (Cl), H (cations and Ag)
- Γ Mixed Common Anion Stock – Cl/SO_4/CO_3 25mg/ml each
- Γ KCl – 1410 µS/cm at 25^{O}C ± 30 µS/cm
- Γ Std Perchlorate expires 1 yr

COMMENTS: _____

2. Quality Control

- Γ **Initial Demonstration** Section 9
- Γ **Low Background**
- Γ **7 replicates** of 25.0 µg/L. **% R = 80 –120% of TV % RSD = < 10%**
- Γ **Second Source % R = 90 – 110%**
- Γ **MDL** – initially determined when a new operator begins work, significant change in background or instrument response. **DO NOT SUBTRACT BLANK VALUES!!**
- Γ **MRL** – Minimum Reporting Level - > 3X MDL or concentration, which would yield a S/N of 5. **MRL must never be lower than the lowest calibration standard**
- Γ **MCT** – Matrix Conductivity Threshold
- Γ If MRL is between 3 – 5 ppb use 25 ppb Perchlorate.
- Γ Prepare a series of solutions with constant Perchlorate conc. And increasing common anions (200, 300, 400, 500 … 1000 mg/L).
- Γ Reference 400 mg/L conductance between 3200 –3700 uS/cm. LFB/200 mg/L mixed common ion should have same A/H and ClO_4 conc.
- Γ Determine % difference (PD) between the average A/H ratios for LFB and each mixed common anion solutions.
- Γ MCT = the matrix conductance where the PD exceeds 20%.
  Plot PD vs. matrix conductance. $r^2 > 0.95$
  - Γ MCT = 20% X (X coefficient) +(constant intercept value)
  - Γ Alternately, MCT conductance level of the highest mixed anion solution which yielded a PD value below the 20% threshold.
  - Γ **MRL** confirmation **% R = 70 – 130%**

Γ Spike soln. at MRL with common anions a conc. est. to generate a conductance near the MCT (within ± 10%).

Γ If fails criteria, lower the MCT by 10% and MRL re-verified.

Γ **LRB = < ½ MRL**

Γ **IPC** (same soln. as MRL confirmation) initially **PD = < 25%**
Repeat MCT if any one fails %**Rec. = 80 – 120%** RT = < 5% shift

Γ **LFB** (use same std as calibration) %**Rec. = 85 – 115%**

Γ **LFM** (spike conc. = or > spl. conc.) %**Rec. = 80 – 120%**

Γ **Dup or LFM dup RPD = ± 15%**

COMMENTS: _____

3. Calibration Section 10

Γ Initial 5 points for concentrations from 4 –400 ppb.
Linearity assumed if **% RSD < 15%** average ration or RF can be used. Use Peak Areas.

Γ Continuing **ICC** – use lowest standard %**Rec. = 75 – 125% CCS** after every 10 and end
%**Rec. = 85 – 115%**

COMMENTS: _____

4. Procedure Section 11

Γ **Conductance of all samples measured. If above the MCT – dilution or pretreatment is performed**.

Γ Dilution done – Check if MRL raised by a proportion equivalent to the dilution.

Γ If Perchlorate found, back calculate. If none found and monitoring is required below the MRL, pretreatment is required.

Γ Pretreatment using BA/Ag/H cartridges same QC limits applied.

Γ LRB and LFB and LFM must be pretreated with any pretreated samples.

COMMENTS:
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5. Data Analysis and Calculations Section 12

Γ RT window within ± 5%

Γ Concentration based on ICAL

Γ Report values between MRL and highest standard.

Γ Reported in ug/L

COMMENTS: _____

6. Notes Section 1, 9, 11

- **For any changes, Section 9.2; 1.2.1 and 11.2.6 must be adhered to**.
- **Maintenance is required when there is reduced peak response and peaks are non-symmetrical**.

COMMENTS: _____