

**Initial demonstration of capability**

- 4020 B.1.a. - Each analyst must run a known standard concentration at least four times and compare limits listed in the method.
  - Keep a copy of the analyst's DOC.
  - Documentation (signed form) that analyst has read and understand all appropriate SOPs and Methods.
  - Recommend backup analyst do this once per year.
  - Only good for the type of instrument you are using at the plant. If you have a backup instrument, you will need another DOC for this instrument.
    - DOCS demonstrate that you are proficient with that instrument.
- **Real people language – each operator running this test needs to analyze 4 samples of  $\text{KMnO}_4$  at a concentration of 0.5 mg/L**
  - **Keep a copy of the analyst's DOC in his personal folder.**

**Method Detection Limit (MDL)**

- 1020 B. 4 – As a starting point for selecting the concentration to use when determining the MDL, use an estimate of five times the estimated true detection level ( $5 \times 0.010 \text{ mg/L} = 0.050 \text{ mg/L}$ ).
  - Ideally, prepare and analyze at least seven (7) portions of this solution over a 3-day period to ensure that the MDL determination is more representative of routine measurements as performed in the laboratory.
  - Ideally use pooled data from several analysts rather than data from one analyst.
- **Real people language – have several operators, who run this test, analyze  $\text{KMnO}_4$  at a concentration of 0.05 mg/L over several days with a total of at least 7 samples**
  - **Joe analyzes 3 samples on Monday**
  - **Bob analyzes 3 samples on Tuesday**
  - **Mary analyzes 3 samples on Wednesday**
- Annual (every 13 months) verification required using data collected within the past 24 months. Include all data from the On-going Data Collections and the initial MDL determination where appropriate (<24 months old). A minimum of 7 data points are required for **both spiked samples and method blanks**.
- Refer to the [MDL Examples and EPA Guidance](#) for complete requirements.
- Refer to document titled "Method Update Rule – Method Detection Limit Math 2019" on [Fleming Training Center website](#) for MDL Calculator

**Initial Calibration Verification**

- 1020 B.11.b. – Perform initial calibration using at least three concentrations of standards for linear curves.
- 4020.B.2.a. – Calibrate initially with at least one blank and three calibration standards.
  - The appropriate linear correlation coefficient for standard concentration-to-instrument response should be greater than or equal to 0.995.
- **Real people language – prepare a set of chlorine or potassium permanganate ( $\text{KMnO}_4$ ) standards in accordance with [Guidance for Secondary Stds use in Calibration 1-15-2016](#) monthly.**

### **Method Blank**

- 1020 B.5.– A reagent blank (method blank) consists of reagent water and all reagents that normally are in contact with a sample during the entire analytical procedure.
- 4020 B.2.d. – Include at least one method blank **daily** or with each batch of 20 or fewer samples, whichever is more frequent.
  - If any method blanks measurements are at or above the reporting level, **take immediate corrective action**. *Refer to the MDL guidance.*
- **Real people language – analyze distilled water as a sample by adding a DPD powder pillow and waiting the 3-6 minutes (or as method requires) before reading**
  - **Target value is less than reporting limit**
    - **Reporting limit will be equal to your MDL**
  - **Run on a 5% basis (see batch size for more information).**

### **Laboratory Fortified Blank**

- 1020 B.6. – A laboratory-fortified blank is a reagent water sample to which a known concentration of the analyte of interest has been added.
  - Sample batch = 5% basis = 1 every 20 samples
  - Use an added concentration of at least 10 times the MDL, less than or equal to the midpoint of the calibration curve.
- 4020 B.2.e. – Calculate percent recovery, plot control charts and determine control limits
- **Real people language – analyze chlorine or potassium permanganate standard at a concentration of 0.5 mg/L**
  - **Run on a 5% basis (see batch size for more information).**

### **Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD)**

- NONE

### **Duplicate –**

- 1020 B.12.f. – Calculate RPD (relative percent difference)
- 4020 B.2.f. – Randomly select routine samples to be analyzed twice.
  - Process duplicate sample independently through the entire sample preparation and analysis.
  - Include at least one duplicate for each matrix type daily or with each batch of 20 or fewer samples.
- **Real people language – on a 5% basis (see batch size for more information) analyze 2 samples for chlorine, after reading one, pour out sample and start with a fresh sample**
  - **For reporting purposes, average sample and duplicate.**
  - **Target value is to get close to the first value and have a small RPD (less than 20%)**

### **Continuing Calibration Verification**

- 1020 B.11.c. – Analysts periodically use a calibration standard to confirm that the instrument performance has not changed significantly since initial calibration.
  - Verify calibration by analyzing one standard at a concentration near or at the mid-point of the calibration range.

- 4020.B.2.b. – Verify calibration by periodically analyzing a calibration standard and calibration blank during a run – typically after each batch of 10 samples and at the end of the run.
  - For the calibration verification to be valid, check standards must not exceed 10% of its true value, and calibration blank results must not be greater than one-half the reporting level
- **Real people language**
  - read **Secondary Standards in accordance with [Guidance for Secondary Stds use in Calibration 10-19-2012](#) daily (day of).**
  - **OR run a chlorine or potassium permanganate standard daily.**

**Control Charts** – 1020 B.13.

- **Real people language**
  - **Create and maintain control charts if you have 20-30 data points within 90 days.**
  - **If you do not meet the above criteria, follow QC Acceptance Criteria below.**

**Corrective Action** - 1020 B.5., B.8., & B.15.

**QC Acceptance Criteria**

- Blanks < Method Detection Limit (MDL)
- LFB  $\pm$  15%
- ICV/CCV  $\pm$  10%
- RPD < 20%
- Reporting Limit = MDL

**Batch Size** –

- For samples that need to be analyzed on a 5% basis or once for every 20 samples follow these criteria:
  - If a permit stated that 3 analyses per week, we would allow for a duplicate to be analyzed at least once per month.
    - Pick a date and be consistent, the 1<sup>st</sup> of every month or the 1<sup>st</sup> Thursday of every month. Mark your calendar!!
  - If a permit stated 5 analyses per week, we would suggest twice a month.
    - Pick a date and be consistent, the 1<sup>st</sup> and 15<sup>th</sup> of every month or the 1<sup>st</sup> and 3<sup>rd</sup> Thursday of every month. Mark your calendar!!

**Calculations** –

- % Recovery for LFB
  - =  $\frac{\text{LFB concentration}}{\text{Expected concentration}} \times 100\%$
- RPD – relative percent differences for duplicates and LFM/LFMD
  - =  $\frac{\text{Difference between sample and duplicate}}{\text{Average of the sample and duplicate}} \times 100\%$