Cyanide Sampling – Fact Sheet

Cyanide sampling is required at many facilities. Following proper technique will help you collect cyanide samples that can be processed by a laboratory. If you sample for cyanide, your industrial waste permit states:

> *"A cyanide composite sample shall consist of a minimum of two samples collected at intervals of 15 minutes or greater within a 24-hour period. Each aliquot shall be collected, treated, and preserved in the field in accordance with 40 CFR 136 and 403 appendix E. Treated aliquots may be collected into a single container and analyzed as one sample."*

Note: an aliquot is a portion of a larger whole.

Tips for proper collection

Cyanide (CN) and Cyanide-Amenable to Chlorination (CN-AC) sampling method

Use the same sampling method for CN and CN-AC sampling.

Have all your materials ready before you begin. The procedure of interference testing through preservation must be completed within 15 minutes of collecting the sample.

1. Select proper bottle
    - Check with your lab for bottle requirements.
        - King County Industrial Waste (KCIW) collects CN in a 500-mL, opaque, high-density polyethylene bottle.

2. Collect sample
    - Use a sampling vessel (**not a composite sampler**) to collect each measured individual aliquot for CN analysis.
    - The sampling vessel can be any clean container.
        - KCIW uses stainless steel sampling pitchers with volume graduations.
    - Sampling must be done separately from any 24-hour composite sample collection for metals or other analytes.

3. Test for chlorine interference

Please contact your lab to find out their preferred method for interference testing and removal.

- Analyze a subsample from the wastewater aliquot for chlorine interference.
- KCIW's method is as follows:
    - Pour a 5-mL subsample into a test tube to test for the presence of chlorine using Lovibrand Powder detection packets. If chlorine is detected, the test tube subsample will turn a shade of pink.
    - If the subsample turns pink, treat the original sample aliquot with 5 drops of sodium thiosulfate ($Na_2S_2O_3$); stir with a clean stainless steel stirrer.
    - Retest a 5-mL subsample of the treated aliquot for the presence of chlorine following the same testing procedure.
    - Repeat the testing and treatment procedure until no chlorine is detected in the sample.

  Note: Once the aliquot has been treated with $Na_2S_2O_3$, it **may not** be used for any other monitoring samples. You may choose to fill any other sample bottles (metals, VOA, etc) **before** $Na_2S_2O_3$ treatment. If you do not remove chlorine, interference may be present during lab analysis for CN and may produce results that are unverifiable.
- Test each aliquot that makes up the final composite sample for chlorine.

4. Test for sulfide

- Chlorine and sulfide cannot be present in the same sample at the same time. You must check sample aliquots for sulfide if chlorine is not present.
    - KCIW tests the sample by putting a drop of the wastewater on a lead acetate test strip. If the strip turns black, sulfide is present and the sample will need to be treated at the lab prior to preservation.
- If you find sulfide in the sample, DO NOT ADD sodium hydroxide (NaOH). Notify your lab immediately that you will be bringing in a sample containing sulfide.

CN or CN-AC composites by grab

1. Confirm the size sample you need

- If you are collecting a CN or CN-AC composite sample, you will need to transfer the necessary volume of the aliquot for each sample grab to the composite sample bottle.
    - For the 500-mL sample container, this would be 125 mL per grab sample for a CN(4) and 250 mL per grab sample for a CN(2).
    - Measure using the volume lines on the stainless steel pitcher or sampling container.

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2. Adjust the pH

- As soon as you add the first aliquot to the final sample container, you must add NaOH to the container to raise the pH to 12 or higher. This will preserve the sample.
    - KCIW uses either NaOH pellets or dropper bottles of NaOH liquid.
- Invert the sample several times once you add the NaOH to mix it.
- Test the preserved sample by putting a small amount of sample water onto a pH test strip to confirm that the sample has a pH of 12 or higher. You can do this by pouring or using a disposable pipette. If the pH is **not** 12 or higher, add additional NaOH until the pH is 12 or higher.
- Check the pH in the final sample container after adding each aliquot. Check the final composite sample for pH within 15 minutes of the collection of the last aliquot.

3. Label the sample

- Label the sample bottle and indicate NaOH preservation for the lab.