Three tap water samples were collected from varying locations across Kamloops, BC: Thompson Rivers University (TRU) S237, a residential house in the Dallas neighbourhood, and a residential house in the Aberdeen neighbourhood.

Before each sample was taken, the faucet head was wiped down with a clean disposable cloth and the water was turned on for 30 seconds before filling the sample bottle.

The TRU sample was reacted immediately and did not require any preservation.

The residential tap water samples were preserved with ~12mg of ammonium chloride, filled to create zero headspace, and stored in a refrigerator immediately. These samples were reacted after four days.

2.3.2 *Swimming Pool Water Samples*

Two samples were collected from saltwater-based pools in the Dallas neighborhood of Kamloops, BC. These samples were preserved with ~12mg of ammonium chloride, filled to create zero headspace, and stored in a refrigerator immediately. These samples were reacted after four days.

2.3.3 *Spa Water Samples*

Two spa samples were collected from chlorine-based spas, one in the Dallas neighbourhood and one in the Upper Sahali neighborhood. Both samples were preserved with ~12mg of ammonium chloride, filled to create zero headspace, and stored in a refrigerator immediately. These samples were reacted after four days.

2.4. *Instrumentation*

The samples and standards were analyzed using the Agilent 7890B GC system paired with the Agilent 5977A MSD system along with a PAL liquid autosampler system.

2.5. *Method development*

May 1, 2024

- Initiated first GC-MS method from *Determination of Haloacetic acids in Bottles and Tap Water Sources by Dispersive Liquid-Liquid Microextraction and GC-MS analysis*
- Labeled as Test-1: 40°C hold for 1min, 25°C/min ramp to 180°C hold for 11min, 30°C/min ramp to 250°C hold for 2min, solvent delay at 2.5min
- Injector at 250°C, ion source at 200°C, carrier gas 50kPa Helium 2.0mL/min, 0.2µL injection volume, two washes with chosen solvent
- AirBlank1 was run on Test-1 to determine any base peaks or interferences

May 3, 2024

- AirBlank2 on Test-1 to determine any changes since last run
- OctanolBlank1 on Test-1 to determine base octanol peaks. This included an octanol rinse instead of an air rinse
- AcetoneBlank1 on Test-1 with octanol rinse since acetone is also used as a solvent
- Developed BakeOut-1 to get rid of residual octanol peaks.
    - 40°C hold for 1min, 50°C/min ramp to 250°C hold for 10min
- AirBlank3 on Test-1 which is where the residual octanol was first seen
- AirBlank4 on BakeOut-1 with an octanol peak at 3.6min.
- AirBlank5 on BakeOut-1
- AirBlank6 on Test-1, the octanol peak was at half-height compared to OctanolBlank1

May 9, 2024

- Developed Test-2, based on Test-1, but used a 6min solvent delay
- AirBlank7 on Test-2
- OctanolBlank2 on Test-2 with octanol rinse
- Prepared an 80ppm octyl chloroacetate stock diluted in acetone at 50mL
    - This was later transferred to a brown glass vial
    - This number was chosen from *Determination of Haloacetic acids in Bottles and Tap Water Sources by Dispersive Liquid-Liquid Microextraction and GC-MS analysis*
- Three standards developed with the stock: 200ppb, 1ppm, and 2.13ppm
    - These standards were diluted with acetone and acetate measured with a glass syringe
- From *Haloacetic acids in swimming pools and spa water* the concentrations of total HAAs ranged from 3.1ppb to 3.83ppb while the pools and spas ranged from 70-3980ppb. This will required a wide range of standards, which will be easiest to manage by having a tap water set and a pool set.
- Some of the smaller standard concentrations will be more accurately made if diluted in a large vessel and then transferred to the GC-MS vial instead of diluted in the vial
- Octyl chloroacetate200ppb on Test-2 with octanol rinse
- Octyl chloroacetate1ppm-1 on Test-2 with octanol rinse
    - There was no noticeable peak other than octanol on 200ppb, so I increased the concentration to hopefully see a larger peak
- Octyl chloroacetate2ppm-1 on Test-2 with octanol rinse
- AirBlank8 on Test-2
- AirBlank9 on BakeOut-1
- AirBlank10 on BakeOut-1
- Brainstormed ideas for program changes to separate the octanol and acetate peaks:
    - Lower the solvent delay to 5 or 5.5 min
    - Use the SIM or Scan & SIM settings
    - Change the temperature or hold times

May 10, 2024

- Took injecting syringe out and cleaned using powered syringe cleaner
- Developed Test-3 which lowered the solvent delay to 5min
- AirBlank11 on BakeOut-1
- AirBlank12 on Test-3
- Developed Test-4 which introduced Scan & SIM with chosen ions of 79 and 95m/z along with changing the hold time of 180°C to 4min along with cutting the final ramp as to never reach 250°C. The End Mass was lowered to 210m/z as octyl chloroacetate has a mass of 207g/mol
- Octyl chloroacetate200ppb-2 on Test-4 with octanol rinse
- Octyl chloroacetate1ppm-2 on Test-4 with octanol rinse
    - Seeing of the peak increases with the increase in concentration
    - Spoiler alert: it did
- Developed Test-5 which lengthened the 40°C hold to 3min
- Octyl chloroacetate200ppb-3 on Test-5 with octanol rinse
    - The peak shifted but did not separate at all
    - The peak height stayed the same as the 1ppm run, indicating significant carryover
- AirBlank13 on BakeOut-1
- AirBlank14 on BakeOut-1
- AirBlank15 on BakeOut-1
    - There was still a strong octanol(unconfirmed) peak throughout the bakeouts, so it was considered to switch the rinse to acetone instead of octanol
- Developed BakeOut-2 which changed the inlet temperature to 240°C
- Removed and rinsed the syringe with acetone
- AcetoneBlank2 on BakeOut-2 with acetone rinse
- AcetoneBlank3 on Test-3 with acetone rinse
- Octyl chloroacetate200ppb4 on Test-4 with acetone rinse
- Developed Test-6 which switched to SIM only with 79 and 95 m/z ions
- Octyl chloroacetate200ppb5 on Test-6 with acetone rinse

May 13, 2024

- AirBlank17 on BakeOut-3
- AirBlank18 on Test-5
- AirBlank19 on Test-4
- AcetoneBlank4 on Test-4 with acetone rinse and solvent delay override at 3 minutes
- AirBlank20 on Test-4
- Octyl chloroacetate200ppb6 on Test-4 with acetone rinse
- AirBlank21 on Test-4
- Made three fresh octyl chloroacetate standards diluted in acetone
    - These are labeled with the date prepped
- Octyl chloroacetate100May13-1 on Test-4 with acetone rinse

    - Actual concentration 106.7ppb
- AirBlank22 on Test-4
- Octyl chloroacetate 200May13-1 on Test-4 with acetone rinse
    - Actual concentration 213.3ppb
- AirBlank23 on Test-4
- Octyl chloroacetate400May13-1 on Test-4 with acetone rinse
    - Actual concentration 426.7ppb
- AirBlank24 on Test-4
- Octyl chloroaceate200ppb-7 on Test-4 with acetone rinse
    - This was prepped the week before
- Octyl chloroacetate200ppb#3-1 on Test-4 with acetone rinse
    - This was prepped on this day to confirm the messy chromatogram from the second 200ppb made
    - Actual concentration 213ppb
- Planned out the order of steps for the simultaneous derivatization and reaction
    - See flow chart on pg15

May 14, 2024

- TFAA was transferred from original packaging into a vial with a septum
    - The liquid was transferred quickly with a disposable pipet
    - A septum lid was secured
    - Using a needle tip on a vacuum hose, the air was removed from the vial
    - Nitrogen from a balloon set-up was introduced
    - The last two steps were repeated another two times
- The Na_2SO_4 and H_2So_4 stock was developed
    - 12.38 g of Na_2SO_4
    - 10.0 mL of H_2SO_4
    - 100.0 mL TOC grade water
- A dry run of the derivatization was performed with no HAAs
    - Sonicated 10 min
    - Rested for 40 min
    - 6 mL of the salt and acid mixture
    - Treated as a reagent blank
    - 1 mL octanol, 30 µL TFAA, 1 mL ethanol
    - Reagent Blank 1
- AirBlank25 on BakeOut-2
- AirBlank26 on Test-4
- ReagentBlank1 on Test-4 with octanol rinse
- Remade the 200ppb and 1ppm stock from the Octyl chloroacetate
    - 200#4: 213ppb
    - 1ppm#2: 1.013ppm
- AirBlank27 on Test-4
- AirBlank28 on BakeOut-2

- AirBlank29 on BakeOut-2
- AirBlank30 on Test-4
- AirBlank31 on BakeOut-2
- AirBlank32 on Test-4
- Octyl chloroacetate200#4-1 on Test-4 with acetone rinse
- AirBlank33 on Test-4
- Octyl chloroacetate1ppm#2-1 on Test-4 with acetone rinse
- AirBlank34 on Test-4
- AirBlank35 on BakeOut-3
- Considered steps for the next session
    - Using dichloroacetic acid as a standard for a later peak that isn't being interfered with the octanol
    - Making standard with the HAA mixture
    - Scaling the standards up
    - Vortexing, shaking, and sonicating the reaction due to the lack of visual cues that the solution was mixing fully

May 28, 2024

- AirBlank36 on BakeOut-3
- AirBlank37 on Bakeout-3
- AirBlank38 on Test-4
- ReagentBlank1 that was run on May 14 had an interesting peak at 5.85min
    - This peak seems to be the octyl chloroacetate peak
    - There should be NO product in the reagent blank, leading me to believe that the TOC grade water was contaminated/wasn't fully decontaminated as it originated as Kamloops tap water
    - This means that other water options must be tested for the reagent blank in order to reduce sample contamination
- AirBlank39 on Test-4
- Mixed a new acid and salt stock
    - 50 mL freshly made TOC grade water from Sharon
    - 6.23g Na_2SO_4
    - 5.0 mL H_2SO_4
- The TFAA septum seemed to be degrading. The top was sticky to the touch and "bubbling" with pressure
    - This should not have happened since the Teflon underside of the septum should be inert to TFAA and should not have reacted
    - This could have been caused by a drop of TFAA getting on to the silicon top of the septum
    - It could also be caused by over-puncturing the septum through evacuating the vial and taking the aliquots out
    - A new lid and septum were attached and the vial was evacuated and filled with nitrogen again

- Reacted Reagent Blank 2
    - 6 mL Fresh TOC acid and salt stock
    - 1 mL octanol, 1 mL ethanol, 30 µL TFAA
    - Handmixed for 40 seconds, sonicated for 10 minutes, water bath 20°C
    - Rested for 1:45 hours
- Developed Test-7 which is based on Test-4 but solvent delay moved to 5.70 min
- AirBlank40 on Test-7
- Mixed another acid and salt stock
    - 50 mL LC-MS grade water
    - 6.26 g Na_2SO_4
    - 5.0 mL H_2SO_4
- Reacted Reagent Blank 3
    - 6 mL LC stock
    - 1 mL octanol, 1 mL ethanol, 30 µL TFAA
    - Handmixed for 40 seconds, sonicated for 10 minutes, water bath 21°C
    - Rested 25 minutes
- Reacted Reagent Blank 4
    - 6 mL LC-MS stock
    - 1 mL octanol, 1 mL ethanol, 30 µL TFAA
    - Sonicated 10 minutes, water bath 22°C
    - Rested 30 minutes
- ReagenBlank3 on Test-7 with octanol rinse
- AirBlank41 on Test-7
- ReagentBlank2 on Test-7 with octanol rinse
- AirBlank42 on BakeOut-3

May 30, 2024

- AirBlank43 on Test-7
- ReagentBlank4 on Test-7 with octanol rinse
- The TFAA vial seemed to be reacting with the new septum even after wiping the lid at the end of the day in case of TFAA droplets getting on top
    - The main suspected cause is that after evacuating and refilling with nitrogen 3 times before taking out the aliquots, the septum is being over-punctured and losing integrity
    - A fix would be putting on a new septum every day after use so that it doesn't degrade will stored
    - Another option is to transfer the TFAA into smaller vials already under nitrogen so that one vial could be used for each day of reactions
        - This could be done in either a glove bag or a glove box in order to avoid having to puncture the vials to evacuate them
- AirBlank44 on BakeOut-3

- On the ReagentBlank2,3, and 4 spectra there is a small peak around 10.074 min that aligns with octyl trichloroacetate
- Developed Test-8, based on Test-4 but with 5.70 minute solvent delay, a ramp from 180°C to 250°C at a rate of 30°C /min and a final hold for 2 minutes
    - This will allow the later eluting products to be seen in the reagent blanks
- AirBlank45 on Test-8
- ReagentBlank4#2 on Test-8 with octanol rinse
- AirBlank46 on BakeOut-3
- Prepared a mock tap water sample from the S237 lab sink
    - 11:03 am, May 30, 2024, 24.5°C
    - Let water run for 30 seconds before sampling
    - 50 mL tap water, 5 mL H_2SO_4, 6.19 g Na_2SO_4
- ReagentBlank2#2 on Test-8 with octanol rinse
- Both 4#2 and 2#2 had a peak of interest at 14.467 minutes which could be an octyl dichloroacetate peak
- When comparing 4#2 and 2#2, it seemed as though 4#2 had smaller peaks and therefore less tap water contamination
- AirBlank47 on BakeOut-3

May 31, 2024

- Building the frame for the glove bag (see attached image)

- Performing dry runs of the TFAA transfer in the glove bag with water
- Major updating within this report (writing day)

June 3, 2024

- AirBlank48 on Test-4
- AirBlank49 on Bakeout-3
- AirBlank50 on Test-8
- Performing dry runs in the glove bag and practicing flushing the nitrogen out with just air
- In the nitrogen filled glove bag, transferred the TFAA to 12 smaller vials for single day uses
- Notes for future work:
    - Do glove bags come with different glove sizes? These gloves were unwieldy

- TFAA vials were stored in enclosed glass bottles in the fridge
- Note: After a month of searching, there seems to be no published literature with a standard mass spectrum of dichloroacetate octyl ester, making the identification of this compound extremely difficult

June 4, 2024

- AirBlank51 on Test-8
- With tap water stock made on May 30th
    - 6mL water stock, 1mL ethanol, 1mL octanol, 30µL TFAA
    - Sonicate 10 minutes, 19.6°C, rested 30 minutes
    - Clear after 15 minutes
- TapWater1 on Test-8 with octanol
    - At 9.260min, there is a possible dichloroacetate octyl ester peak as NIST would define the mass spectrum as dichloro acetic acid decyl ester, which is just two carbons longer than the wanted compound. Since NIST does not have the octyl ester, this decyl ester could be a good hint towards identifying the dichloro peak.
- AirBlank52 on Test-8
- Created a fresh tap water stock
    - From sink in S237, 10:29am
    - A total of 5 minutes from sampling to beginning the reaction
    - Stock: 50mL water, 5.4mL H_2SO_4, 6.21g Na_2SO_4
    - 6mL stock, 1mL ethanol, 1mL octanol, 30µL TFAA
    - Sonicate 10 minutes, 19.6°C, rest for 20 minutes
- TapWater2 on Test-8 with octanol rinse
    - At 6.156min, there is an unknown peak of interest
- AirBlank53 on Test-8
- AirBlank54 on Bakeout-3
- Developed Test-9 which is based on Test-8 but sets the SIM ions to 50, 84, and 119 which are M^+-157 for each of the chlorinated molecules as recommended in the literature. Also drops lowest m/z mass to 35 from 40
- AirBlank55 on Test-9
- TapWater2-2 on Test-9 with octanol rinse
- AirBlank56 on Test-8
- AirBlank57 on Bakeout-3
- Developed Test-10 which is based on Test-9 but changes SIM ions into three groups
    - MCAA (5.70min) 79,95
    - DCAA (6.30min) 48,76
    - TCAA (11.5min) 36,110,121
- TapWater2-3 on Test-10 with octanol rinse
- AirBlank58 on Bakeout-3

June 20, 2024

- Developed Test-11 which is based on Test-10 but changes the group 3 SIM time to 8.00 minutes
- AirBlank59 on Test-11
- Brainstormed possible next steps
    - Making a octyl dichloroacetate standard
        - This would have to be reacted like the reagent blanks since the compound we have is the acetic acid variation
    - Retest the octylchloro acetate standards
    - Fiddle with the volume of water needed for the new, smaller vials
- Mixed a dichloroacetic acid stock to 62.5ppm
    - 2.0 µL 99.9% dichloroacetic acid
    - Diluted to 50.0 mL with the "New TOC Water" Sharon prepped
- Reacted dichloro-1
    - 1 mL ethanol, 1 mL octanol, ~50 µL TFAA, 100 µL dichloroacetic acid, 6 mL new TOC acid and salt stock (prepared May 28)
    - Sonicated for 10 minutes, rested for 30 minutes
    - The TFAA vials seemed to be contaminated
        - When using an autosampler vial of TFAA, the septa collapsed into the vial when punctured by the nitrogen balloon
            - This vial was used after being exposed to air for ~7 minutes
        - The conical reaction vials of TFAA seemed to still have strong septa, but the beige top turned black and was impossible to pierce with the nitrogen balloon

- AirBlank60 on BakeOut-3
- AirBlank61 on Test-8
- Dichloro-1 on Test-8 with octanol rinse
- AirBlank62 on Bakeout-3
- AirBlank63 on Test-8
- Prepared Dichloro-2
    - 1 mL ethanol, 1 mL octanol, ~40 µL exposed TFAA, 50 µL dichloroacetic acid, 6 mL new TOC acid and salt stock (prepared May 28)
    - Sonicated 10 minutes, rested 30 minutes
    - 0.52ppm
- Dichloro-2 on Test-8 with octanol rinse

    - When comparing Dichloro-1 and Dichloro-2 via their percent report peak area, three possible peaks stood out for have a reasonable increase in area to increase in concentration: 6.039, 6.286, 14.463 min
- AirBlank64 on BakeOut-3
- Brainstormed for the next session
    - Create two new dichloro standards for 0.75 and 2.00 ppm to compare to the 0.5 and 1.0 ppm from this day
    - Transfer the TFAA from the conical reaction vials to autosampler vials in the glove bag

June 21, 2024

- AirBlank65 on Test-8
- Dichloro2-2 on Test-8 with octanol rinse
- AirBlank66 on Bakeout-3
- AirBlank67 on Test-8
- Dichloro1-2 on Test-8 with octanol rinse
- AirBlank68 on Bakeout-3
- AirBlank69 on Test-8
- TFAA in conical reaction vials were transferred to autosampler vials under nitrogen in a glove bag
- Prepared Dichloro-3
    - 1 mL ethanol, 1 mL octanol, 30 µL (freshly transferred) TFAA, 60 µL dichloroacetic acid, 5 mL new TOC acid and salt stock (prepared May 28)
    - 0.75 ppm
    - Sonicate 10 minutes, rest 30 minutes
- Prepared Dichloro-4
    - 1 mL ethanol, 1 mL octanol, 30 µL (freshly transferred) TFAA, 160 µL dichloroacetic acid, 5 mL new TOC acid and salt stock (prepared May 28)
    - 2.00 ppm
    - Sonicate 10 minutes, rest 30 minutes
- Dichloro3-1 on Test-8 with octanol rinse
- AirBlank70 on Bakeout-3
- AirBlank71 on Test-8
- Dichloro4-1 on Test-8 with octanol rinse
- AirBlank72 on Bakeout-3
- Dichloro4-2 on Test-8 with octanol rinse
- AirBlank73 on BakeOut-3
- On dichloro3, there's a peak of interest at 6.847 min
- There is no peak at 6.286 in either of the dichloro4 runs, suggesting that peak is not dichloro on the other concentrations' runs
- Brainstorming for next week
    - Changing the ions that the sim and scan program searches for
    - Doing only sim to help isolate the peaks

- Making new dichloro standards all in one day
- Cleaning the TFAA syringe as a piece of infected septa could have gotten lodged in the needle
- Creating a stock using the mix of 6 HAAs

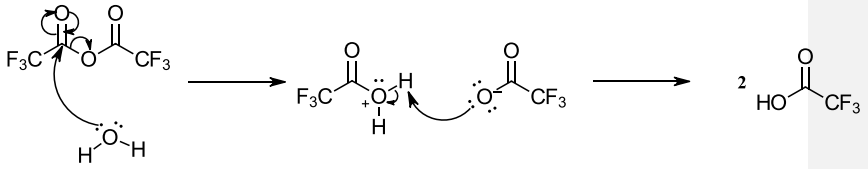
June 26, 2024

- Bakeout-4 is identical to Bakeout-3
  - This was saved after the GCMS app would not update the type of PAL sampler installed (the program thought that a headspace syringe was installed, not the liquid syringe)
- AirBlank74 on BakeOut-4
  - Did not run due to sampler error
- AirBlank75 on BakeOut-3
- Test-12 is based on Test-9 but with SIM only and the ions are changed to 48 and 76 m/z as suggested by literature
- AirBlank76 on Test-12
- Dichloro2-3 on Test-12 with octanol rinse
- AirBlank77 on Bakeout-3
- Dichloro3-2 on Test-12 with octanol rinse
- AirBlank78 on BakeOut-3
- Dichloro1-3 on Test-12 with octanol rinse
- AirBlank79 on BakeOut-3
- Dichloro4-3 on Test-12 with octanol rinse
- AirBlank80 on BakeOut-3
- When I compared the four SIM chromatograms, four peaks stood out
  - 6.952, 7.05, 7.8, 12.56
  - None of these worked as the ratio of ions did not match the mass spectrum for dichloro that was found in literature
- On Dichloro1-3, there was a promising peak at 6.133 which was the only peak with 76 > 48 m/z, which is the ratio that was needed. While the ratio would preferably be 2:1, this is the closest yet

June 27, 2024

- AirBlank81 on BakeOut-3
- Developed Test-13 based on Test-12
  - Changed it to SIM&Scan
- AirBlank82 on Test-13
- When opening the jar that contained the vials of TFAA, there was an intense vinegar odour, which indicated that the TFAA was exposed to air/ leaked
  - This leads to me not being able to complete and reactions as the TFAA is a necessary reagent
- The new TFAA will be arriving mid to late July, which leaves lots of spare time to write and research

    - With the spare time, I may decide to try a version of the EPA method to compare directly to my method
- Wrote out a proposed mechanism for the reaction of TFAA in water



June 28, 2024

- Spent most of my time researching versions of the EPA method
    - Wanted to find some that adapted to GC-MS
- Started a source table to compile and summarize sources for ease of information searching later on

July 2, 2024

- Compiled a table of three methods based on the EPA 552.3 but adapted to GC-MS
    - Listed relevant information for each method to determine the best one to follow
    - Due to it's nearness to the EPA 552.3 method, *Validation and application of a GC–MS method for the determination of haloacetic acids in drinking water* was the most favourable out of the three options
    - This allows for a direct comparison between the standard EPA method and my new method
- Nearly finished the introduction
    - Just minor editing and source formatting left

July 3, 2024

- Wrote out procedure for extraction and derivatization from *Validation and application* without added standards
    1. In an extraction tube, add 20 mL of the selected water
    2. Add ~1 mL of sulfuric acid until the pH is 0.5
    3. Add 9 g anhydrous Na_2SO_4
    4. Add 2 mL MTBE and shake for 3 min
    5. Led stand until separated
    6. Draw 1.5 mL of the top organic layer and dispense into a clean container
    7. Add 1.5 mL H_2SO_4:CH_3OH solution to the organic layer
    8. Quickly close and place in a heating bath at 50.0 °C for 2 hours
    9. Cool in an ice bath
    10. Add 4.0 mL of 1.0 M Na_2SO_4 and vortex for 3 minutes
    11. Remove all the top organic layer along with some of the aqueous layer and transfer to a new container
    12. Add 1.0 mL saturated sodium bicarb solution to the organic layer

13. Vortex for 30 seconds
14. Collect top layer for analysis

- Collected and prepared glassware and reagents
- Created methyl dichloroacetate stock at 82.86ppm in MTBE
- Developed Test-14, which is the EPA method
- AirBlank83 on Test-14
- MTBE-1 on Test-14 with MTBE rinse
- Planned out methyl dichloroacetate standards for next week
- AirBlank84 on Bakeout-3

July 10, 2024

- AirBlank85 on Test-14
  - There seems to be an octanol peak near 20ish minutes
  - This makes no sense as many air blanks and bakeouts have been run since octanol was last used
- AirBlank86 on Test-14
- Performed a dry run of the EPA reaction to create a reagent blank
  - 20 mL LC-MS grade water, 8.98 g Na_2SO_4
  - prepared 10 mL of 1:9 H_2SO_4:methanol
  - prepared 25 mL 1.0M Na_2SO_4 in LC-MS grade water
  - prepared 25 mL saturated sodium bicarbonate with 3.51 g sodium bicarb
- AirBlank87 on Test-14
  - Trying to eliminate the octanol peak
- EPA Reagent-1 on Test-14 with MTBE rinse
  - Determined that the shoulder from 2.5-4.0 min is MTBE
- AirBlank88 on BakeOut-3
- AirBlank89 on Test-14

July 11, 2024

- AirBlank90 on Test-14
- Prepared methyl dichloroacetate standards in 1.5 mL autosampler vials up to volume with MTBE
  - 20 µL methyl dichloroacetate stock, 1.1ppm
  - 15 µL methyl dichloroacetate stock, 0.83ppm
  - 10 µL methyl dichloroacetate stock, 0.55ppm
  - The stock was left out of the refrigerator for a day, so there is a chance that is degraded and makes the results off
- Developed Test-15 based on Test-14, changed to SIM & Scan with 59, 83, 85, and 111
- Methyl dichloroacetate0_5-1 on Test-15 with MTBE rinse
  - With the length of this temperature program reaching over 35 minutes, it will be hard to adjust concentrations in the same day as it would take ~2 hours to compare two runs

- AirBlank91 on BakeOut-3
- AirBlank92 on Test-15
- Methyl dichloroacetate0_8-1 on Test-15 with MTBE rinse
- AirBlank93 on Bakeout-3
- Methyl dichloroacetate1-1 on Test-15 with MTBE rinse
- Performed a run with tap water from S237 using the EPA method
    - Let tap run for 30 seconds before sampling at 1:40pm
    - 20 mL tap water, 9.05 g Na_2SO_4
- AirBlank95 on Bakeout-3
- AirBlank96 on Test-15
- Brainstormed for tomorrow
    - Test a methyl dichloro standard with Chris's program and see if the retention time is the same as his to confirm the presence of the product

July 12, 2024

- AirBlank97 on Test-15
- EPA-TapWater-1 on Test-15 with MTBE rinse
- AirBlank98 on Bakeout-3
- AirBlank99 on Chris's Method-1
- Chris's method-2 changes solvent delay to 2 min
- AirBlank100 on Chris's Method-2
- Chris methyl dichloroacetate1-1 on Chris-2 MTBE rinse
- Chris's method-3 changes solvent delay to 2.5 and SIM only for 59, 83, 85, 87
- Chris methyl dichloroacetate1-2 on Chris-3 MTBE rinse
    - The peak was very clear at the anticipated 5.5 min for DCAA-m
    - This proves the presence of DCAA-m in the standard
    - With a stronger standard, it may be more noticeable on Test-15
    - If the stronger standard still doesn't work, I can switch Test-15 to SIM only and consider using Chris's ions instead of the ones I selected
- AirBlank101 on bakeout-3
- With the 80ppm methyl dichloroacetate stock:
    - Made a 9.94ppm standard (180 µL)
- AirBlank102 on Test-15
- Methyl dichloroacetate10-1 on Test-15 with MTBE rinse
    - Confirms the DCAA-m peak at ~8.5min
- AirBlank103 on Bakeout-3
- Brainstormed for the next week
    - Create a SIM only program
    - Cut off program around 12 minutes
    - Run standards on new program
    - Run water sample on new program

July 17, 2024

- The GC-MS was glitching and not registering the syringe volume
    - This required Test-15 to be resaved as an identical Test-16
- AirBlank104 on Test-16
- Developed Test-17
    - Based on Test-16
    - 40°C hold 10 min, ramp 2.5°C/min to 50°C
    - This is to shorten the length of the program since the DCAA-m peak is at 8.5 min
- Methyl dichloroacetate1-2 on test-17 with MTBE rinse
- Developed Test-18
    - Based on Test-17
    - SIM only
- AirBlank105 on Test-18
- Methyl dichloroacetate1-3 on Test-18 with MTBE rinse
- Test-19
    - Based Test-18 but 3 minute solvent delay
    - To make it easier to identify product peak without solvent shoulder
- AirBlank106 on Test-19
- Methyl dichloroacetate0_8-2 on Test-19 with MTBE rinse
- AirBlank107 on Test-19
- Methyl dichloroacetate0_5-2 on Test-19 with MTBE rinse
- AirBlank108 on Test-19
- Through percentage reports, the three DCAA-m trials on Test-19 showed a prominent increase in both peak head and peak area as concentration increased
    - This confirms that the temperature program with those set ions can consistently detect and measure DCAA-m
- EPATapWater-2 on Test-19 with MTBE rinse
    - Confirmation of
- AirBlank109 on Test-19
- AirBlank110 on Bakeout-3

July 18, 2024

- AirBlank111 on Bakeout-3
- Developed Test-20 based on Test-15 but changed solvent delay to 3 minutes
    - SIM Groups:
        - MCAA @3.00 min 59, 64, 77 m/z
        - DCAA @8.30 min 59, 83, 85, 111 m/z
        - TCAA @10.0 min 59, 117, 119 m/z
- AirBlank112 on Test-20
- EPATapWater-3 on Test-20 with MTBE rinse
    - Peak of interest at 14.4 min that could be TCAA
- Created 50 mL of 20ppm HAA mix stock in MTBE from 2000ppm solution

- Diluted HAA stock in 25 mL volumetric flasks with MTBE to the following concentrations
    - 1 ppm, 0.4 ppm, and 9.6 ppm
    - These were all tossed as I had forgotten that they had to be derivatized
- AirBlank113 on Test-20
- EPA HAA0_4-1 on Test-20 with MTBE rinse
    - This was the plain diluted HAA, and therefore shall be ignored
- AirBlank114 on test-20
- Remade standards properly with reaction for 0.5ppm and 0.25 ppm
    - Stored in autosampler vials in freezer

July 19, 2024

- AirBlank115 on Bakeout-3
- AirBlank116 on Bakeout-3
- Developed Test-21 based on Test-21 with SIM only
- AirBlank117 on Test-21
- EPA HAA0_25ppm-1 on Test-21 with MTBE rinse
- AirBlank118 on Bakeout-3
- AirBlank119 on Bakeout-3
- AirBlank120 on Test-21
- Created three new standards from the 20 ppm HAA stock
    - 100 ppb, 50ppb, and 25 ppb
    - Fully reacted
- Refilled the 1M Na_2SO_4 solution in LC-MS water
    - 3.55g
- EPA HAA 0_5ppm-1 on Test-21 with MTBE rinse
- AirBlank121 on Bakeout-3
- AirBlank122 on Test-21
- EPA HAA0_1ppm-1 on Test-21 with MTBE rinse
- AirBlank123 on Bakeout-3

July 24, 2024

- AirBlank124 on Bakeout-3
- AirBlank125 on Test-21
- Created a 5ppm standard using the 20ppm HAA stock in MTBE
- EPA HAA 25ppb-1 on Test-21 with MTBE rinse
- AirBlank126 on Bakeout-3
- AirBlank127 on Test-21
- EPA HAA 50ppb-1 on Tes-21 with MTBE rinse
- AirBlank128 on Bakeout-3
- AirBlank129 on Test-21
- EPA HAA 5ppm-1 on Test-20 with MTBE rinse
- EPA HAA 5ppm-2 on Test-20 with MTBE rinse

- AirBlank130 on Bakeout-3
- Brainstormed for tomorrow
    - Run tap water on a SIM only program for the EPA method
    - Create a stock for the dual derivatization and extraction reaction
    - Create standards for the above reaction

July 25, 2024

- AirBlank131 on bakeout-3
- AirBlank132 on Test-21
- EPA TapWater-4 on Test-21 with MTBE rinse
- Created HAA stock mix
    - 25 mL volumetric flask
    - Diluted in acetone
    - 0.35 mL for 28ppm
- AirBlank133 on Bakeout-3
- Developed Test-22 based on Test-8 with added SIM and Scan ions of 79, 95, 48, 76, 121, 123, 139, 36, 110, 0127, 129, 131, 120, 122, 173
- AirBlank134 on Test-22
- Made a 35 ppb standard but did not mix it thoroughly, labeled as no mix
- AirBlank135 on Test-22
    - Previous blank didn't look clean enough
- HAA no mix 35 ppb-1 on Test-22 with octanol rinse
- AirBlank136 on Test-22
- Created 0.49, 2.8, and 5.0 ppm standards
- AirBlank137 on Test-22
- HAA EPA

Results:

3.1 MS Peak Identification

Compound Name	Elution Time (min)
Octyl chloroacetate	
Octyl bromoacetate	
Dichloroacetic acid octyl ester	
Trichloroacetic acid octyl ester	

3.2 Finalized Reaction Sequence