



September 17, 1984

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Analytical and
Consulting Chemists

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- NPDES
- USDA
- USEPA

Ms. Elizabeth Betz
Quality Control Laboratory
NREAD
Marine Corp Base
Camp Lejeune, North Carolina 28542

Dear Ms. Betz:

Please find attached EPA guidelines for sampling for MAXIMUM Total Trihalomethane Potential. I believe you have already received adequate sample bottles for triplicate sampling. The reagent in the bottles is Calcium Hypochlorite. This will insure a chlorine residual at the end of the storage period. Be sure to incubate samples in an area that is free from vapors of chlorinated solvents. When checking for a residual chlorine after the 7 day incubation, use the one sample of the triplicate which has the largest air bubbles or is otherwise lease suitable for subsequent analysis.

Should you have any questions, please contact me at (919) 787-3061.

Sincerely,

Bruce A. Babson
Sr. Staff Chemist

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the exact retention time for the trihalomethane.

ATT = attenuation factor.

9.5 Report the results obtained from the lower limit of detection estimates along with the data for the samples.

10. Precision and Accuracy

10.1 Single lab precision and accuracy. The data in Table 2 were generated by spiking organic-free water with trihalomethanes as described in 5.9. The mixtures were analyzed by the analyst as true unknowns.

References

1. MIEURE, J.P., "A Rapid and Sensitive Method for Determining Volatile Organohalides in Water," *Journal AWWA*, 69, 60, 1977.
2. REDING, R., et al. "THM's in Drinking Water: Analysis by LLE and Comparison to Purge and Trap", *Organics Analysis in Water and Wastewater*, STP 686 ASTM, 1979.
3. "Handbook for Analytical Quality Control in Water and Waste Water Laboratories," Analytical Quality Control Laboratory, National Environmental Research Center, Cincinnati, Ohio, June 1972.
4. BUDE, W.L., J.W. EICHELBERGER, "Organic Analysis Using Gas Chromatography-Mass Spectrometry," Ann Arbor Science, Ann Arbor, Michigan, 1979.
5. "The Analysis of Trihalomethanes in Finished Water by the Purge and Trap Method," Environmental Monitoring and Support Laboratory, Environmental Research Center, Cincinnati, Ohio, 45268, May 15, 1979.
6. RICHARD J.J.; G.A. JUNK, "Liquid Extraction for Rapid Determination of Halomethanes in Water," *Journal AWWA*, 69, 62, January 1977.
7. BRASS, H.J., et al., "National Organic Monitoring Survey: Sampling and Purgeable Organic Compounds, Drinking Water Quality Through Source Protection," R.B. Pojasek, Editor, Ann Arbor Science, p. 398, 1977.
8. WHITE, L.D., et al. "Convenient Optimized Method for the Analysis of Selected Solvent Vapors in Industrial Atmosphere," *AIHA Journal*, Vol. 31, p. 225, 1970.
9. KOPFLER, F.C., et al. "GC/MS Determination of Volatiles for the National Organics Reconnaissance Survey (NORS) or Drinking Water, Identification and Analysis of Organic Pollutants in Water," L.H. Keith, Editor, Ann Arbor Science, p. 87, 1976.

Determination of Maximum Total Trihalomethane Potential (MTP)*

The water sample used for this determination is taken from a point in the distribution system that reflects maximum residence time. Procedures for sample collection and handling are given in EMSL Methods 501.1 and 501.2. No reducing agent is added to "quench" the chemical reaction producing THMs at the time of sample collection. The intent is to permit the level of THM precursors to be de-

pleted and the concentration of the THMs to be maximized for the supply being tested.

Four experimental parameters affecting maximum THM production are pH, temperature, reaction time and the presence of a disinfectant residual. These parameters are dealt with as follows:

Measure the disinfectant residual at the selected sampling point. Proceed only if a measurable disinfectant residual is present. Collect triplicate 40 ml water samples at the pH prevailing at the time of sam-

*40 C.F.R. Part 141, Appendix C, Part III.

pling, and prepare a method blank according to the EMSL methods. Seal and store these samples together for 7 days at 25° C or above. After this time period, open one of the sample containers and check for disinfectant residual. Absence of a disinfectant

residual invalidates the sample for further analyses. Once a disinfectant residual has been demonstrated, open another of the sealed samples and determine total THM concentration using either of the EMSL analytical methods.

Method for Volatile Chlorinated Organic Compounds in Water and Wastewaters*

NOTE.—Most of the compounds listed in Para. 1.2 of the following method are determined by Method 514, 15th Edition of *Standard Methods for the Examination of Water and Wastewater*, but the method may be useful for the benzene derivatives.

1. Scope and Application

1.1 This method covers the determination of various chlorinated organic compounds in water and wastewater.

1.2 The following chlorinated organic compounds may be determined individually by this method:

Benzylchloride
Carbon tetrachloride
Chlorobenzene
Chloroform
Epichlorohydrin
Methylene chloride
1,1,2,2-Tetrachloroethane
Tetrachloroethylene
1,2,4-Trichlorobenzene
1,1,2-Trichloroethane

2. Summary

2.1 If the sample is turbid, it is initially centrifuged or filtered through a fiber glass filter in order to remove suspended matter.

*"Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater" (INTERIM, Pending Issuance of Methods for Organic Analysis of Water and Wastes, September 1978). Environmental Protection Agency, Environmental Monitoring and Support Laboratory (EMSL).

A three to ten microliter aliquot of the sample is injected into the gas chromatograph equipped with a halogen specific detector. The resulting chromatogram is used to identify and quantitate specific components in the sample. Results are reported in micrograms per liter. Confirmation of qualitative identifications are made using two or more dissimilar columns.

3. Interferences

3.1 The use of a halogen specific detector minimizes the possibility of interference from compounds not containing chlorine, bromine, or iodine. Compounds containing bromine or iodine will interfere with the determination of organochlorine compounds. The use of two dissimilar chromatographic columns helps to eliminate this interference and, in addition, this procedure helps to verify all qualitative identifications. When concentrations are sufficiently high, unequivocal identifications can be made using infrared or mass spectroscopy. Though non-specific, the flame ionization detector may be used for known systems where interferences are not a problem.

3.2 Ghosting is usually attributed to the history of the chromatographic system.

VOLATILE CHLORIN.

Each time a sample is injected, small amounts of various compounds are sorbed on active sites at the head of the column. These compounds of water tend to stick to the column, resulting in non-replicable peaks. This displacement of the phenomenon normally observed in the analysis of a series of high concentrations is followed by a ghosting. The system should be flushed before each peak is injected. The system should be flushed by injecting distilled water. If identical to the sample, ghosting occurs. Corrective measures should be taken as required in the order list.

- 1) Multiple flushes
- 2) Clean or replace liner
- 3) Replace the chromatogram

4. Apparatus and Materials

4.1 Gas Chromatograph—The gas chromatograph programmed oven temperature and glass-lined injector should be equipped with a support and heated transfer line attachment to the detector.

4.2 Detector Option

4.2.1 Microcoulometer

4.2.2 Electrolytic Cell

4.2.3 Flame Ionization

4.3 Recorder—Potentiometer recorder (10 in) computer.

4.4 Syringes—1 µg.

4.5 BOD type bottle vials sealed with Teflon septa.

4.6 Volumetric Flasks—10 ml.

4.7 Syringe—Hypodermic (30 ml).

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SAMPLE COLLECTION INSTRUCTIONS FOR THM ANALYSIS

1. You have received two (2) sampling vials for each sample that you have requested Grainger Laboratories, Inc. to analyze. We need both vials filled properly in order to assure you of the quality of the analytical results.
2. Examine each vial. There is a screw cap, liner, sample vial, and a small crystal of a preservative in the vial. DO NOT remove this crystal.
3. Examine the liner. You can see that there are two (2) distinct sides. The thicker, darker colored side should be up. The thinner, shiney side is the teflon coating and should be down. Please refer to Figure I.
4. Carefully fill the vial so as not to lose the crystal of preservative. The sample vial should be over filled as shown in Figure 2. Examine the filled vial carefully. Note if there are any bubbles trapped on the sides of the vial. If there are bubbles, they may be removed by tapping the sides of the vial. Refill the vial as shown in Figure 2 if necessary.
5. Refer to Figure 3. Properly placing the liner on the vial is the most critical step. Be sure that the teflon side of the liner is down! To insure that no bubbles are trapped in the sample, the liner is placed on the edge of the vial. A slight upward angle is helpful. The liner is now smoothly and sharply slid into place on the vial. The over filled water bulge is displaced and no air bubbles are trapped in the sample. Now replace the screw cap and tighten finger tight. Invert the vial to check for any air bubbles. If there are any present, remove the screw cap and liner and over fill the vial with sample once again. Do not pour out the sample as you will lose the preservative which has dissolved. Repeat step 5 until no air bubbles are trapped in the sample. Patience, after a few attempts you will get the hang of it.

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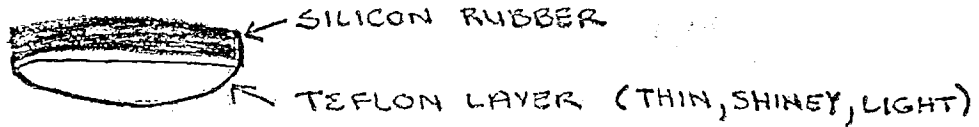
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FIGURE 1



CAP LINER

FIGURE 2

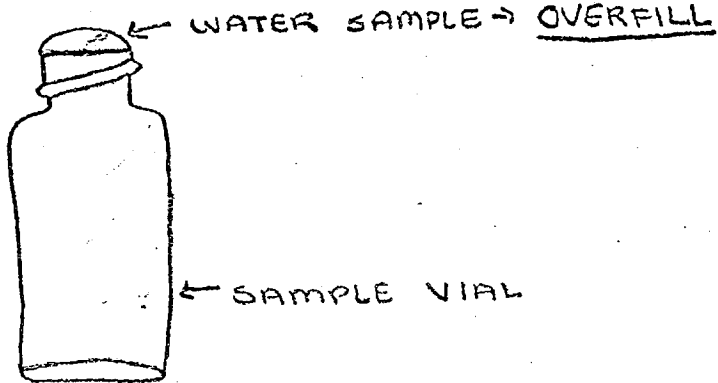


FIGURE 3

