## FORMATION AND MEASUREMENT OF TRIHALOMETHANES IN DRINKING WATER

by

Alan A. Stevens and James M. Symons

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### INTRODUCTION

Because of findings concerning the carcinogenicity of chloroform and the confirmation of the ubiquity of chloroform and other trihalomethanes in chlorinated drinking water, many purveyors of potable water are interested in sampling their product to determine the extent of their individual chloroform problems and resolve them when possible. Additionally, as a direct result of the announced EPA policy regarding the initiation of a Voluntary Nationwide Chloroform Reduction Program, other water utilities are anticipated to attempt to reduce, through modification of the treatment process, concentrations of chloroform reaching the consumer.

In these treatment-modification and surveillance programs, difficulties often arise concerning both the factors that must be considered in selecting modifications of treatment and what should be considered when selecting sampling and analysis techniques to best evaluate the full extent of the problem or the success or failure of the efforts to reduce that problem. The physical and chemical factors controlling production of chloroform and the influence of these factors on the concentrations of chloroform and other trihalomethane (THM) species that are observed in a sample at the time of analysis must be understood.

In addition to physical and chemical considerations, adequate bacteriological monitoring of finished waters during chloroform-reduction programs must be included. Any research program in which disinfection practice is a variable requires careful attention on the part of the utility operators to ensure that water of adequate microbiological quality reaches the consumer.

<sup>\*</sup>This paper is a summary of information that is presented in considerably more detail in the three publications listed under references, below. Those three references include all appropriate supporting literature citations, and the reader is referred to them for additional reading. For that reason, individual literature citations do not appear in this paper.

# FACTORS INFLUENCING THM FORMATION THAT AFFECT TREATMENT STRATEGIES

### General

The formation of trihalomethanes during chlorination of drinking water now seems to be well accepted to result from a complicated mechanism of attack by aqueous halogen species on natural aquatic humic substances (humic and fulvic acids) and not usually significantly from the sources of industrial water pollution. Thus chloroform results from the generalized reaction,

aqueous chlorine + "precursor" (humic Acids) >

### chloroform

This occurs to some extent in any water-treatment plant where chlorination for disinfection is practiced. The reaction is not instantaneous and occurs over a period of a few days until either chlorine or precursor is exhausted. In the presence of natural bromide, the reaction products include some mixed halogen trihalomethane species (bromodichloromethane, dibromochloromethane) and bromoform. This occurs in most chlorinated drinking water, even where bromide concentrations in the source water are small. Iodine-containing species have also been observed, presumably because of the presence of natural iodide. Because the chemical reactions for formation of these bromine- and iodine-containing trihalomethanes are probably mechanistically similar to those for formation of chloroform, the trihalomethanes including chloroform, can be discussed as a group for treatment evaluations.

Design of the most effective treatment strategy depends on a good knowledge of factors influencing trihalomethane formation. Two factors, however, that have a strong influence on trihalomethane concentrations over which the water treatment plant operator has little or no control under most circumstances are temperature and  $Br^-$  or  $I^-$  concentrations.

#### Temperature

Figure 1 clearly demonstrates the positive effect of increasing temperature on trihalomethane formation upon chlorination of Ohio River water in the laboratory. A corresponding seasonal variation is noticed at a water utility using that same source and has been shown to be largely a temperature effect. Thus treatment problems become more acute during seasons of higher ambient air temperature causing higher water temperature during treatment and distribution.

### Bromide and Iodide Concentration

Bromide and iodide ions are oxidized by aqueous chlorine to species capable of participating in organic substitution reactions resulting in the formation of pure- and mixed halogen trihalomethanes. Bunn, et al. of USEPA, Kansas City, first confirmed one of the suspicions of Rook in the Netherlands that this could occur in aqueous systems when they chlorinated Missouri River water in the presence of added fluoride, bromide, and iodide



FIG.1 EFFECT OF TEMPERATURE ON CHLOROFORM FORMATION

and observed the formation of all ten possible chlorine-, bromine-, and iodine-containing pure- and mixed halogen trihalomethanes. On a theoretical basis, fluorine substitution was not expected and was not observed. To date, at least six of these species have been found in finished drinking water (chloroform, bromodichloromethane, dibromochloromethane, bromoform, dichloriodomethane, and bromochloroiodomethane).

Figure 2 illustrates the results of work conducted in our laboratory on the effect of added bromide on the ratio of trihalomethanes produced during reaction of aqueous chlorine with humic acids. Note that bromine substitution is favored over chlorine even though chlorine is in large excess compared with the initial bromide. Additionally, the total molar yield of trihalomethanes increases with increasing bromine substitution. This was also observed when pure aqueous bromine was reacted with the humic acid under the same conditions as aqueous chlorine. Thus, bromine competes more effectively than chlorine for active sites on the humic acid precursor molecule, perhaps mechanistically by way of a faster substitution reaction rate. This effect is so pronounced as to dramatically increase total haloform yields where bromide is present. Indeed, similar increases in total haloform yield have been reported to occur on chlorination of a bromide spiked natural water and more importantly at a water treatment plant in the USA where sea water intrusion was temporarily responsible for increases in bromide (Fig. 3, Lange, et al., Contra Costa, CA). Thus, much more complete control of trihalomethane precursor, as one method of meeting proposed USEPA drinking water standards, is necessary when significant concentrations of bromide are present in the source water.

### Effect of pH

Increasing the pH of the water being treated has been shown by numerous workers to dramatically influence rates of formation of haloforms during water treatment (Fig. 4). Control of pH during treatment before chlorination, such as recarbonation in a lime softening system, has been used in attempts to control haloform formation.

The increase of trihalomethane formation rate with pH was expected because the classical haloform reaction is base catalyzed; however, this explanation is likely to be an oversimplification where rather complex humic acid structures are involved. Simple methyl ketones, models for the haloform reaction, have been shown to react too slowly to account for trihalomethane formation under most drinking water treatment conditions. Figure 5 shows the results of attempts to react chlorine at pH 7 with simple acetyl compounds (acetone, acetaldehyde, and acetophenone), when these compounds were spiked at 5 umol/L into raw- and granular activated carbon (GAC) filtered water. Trihalomethanes were not produced at rates significantly higher than those for the blank samples.

Figure 6, however, shows that at higher pH values, the simplist methylketone, acetone, reacts at a much higher rate, and this class of compounds could become a significant source of precursor in those pH ranges.











The complex humic structure therefore must have more active groups than the simple methyl ketones for chlorine substitution to account for reactivity at pH 7 with the possibility of less active (actyl) groups becoming more significant at higher pH, increasing reaction rate and yield.

An alternate explanation for the effect of pH on rate and yield with humic acid precursor has been suggested, by Christman, however (personal communication). The macromolecule may simply be "opening up" by mutual repulsion of the negative changes at high pH increasing the availability of additional reactive sites on that molecule.

### Characteristics and Concentration of Precursors

In artifical systems, increasing the concentration of humic acid precursor in the presence of excess chlorine with otherwise constant reaction conditions causes haloform yields to increase in direct proporation to the humic acid dose (Fig. 7). Although at similar TOC concentrations, humic acids and natural water have been shown to result in similar THM yields (Fig. 8), from supply to supply, only crude relationships have been found between organic carbon concentrations and trihalomethane yields. Similarly the relationship is not perfect when water at various stages of treatment are examined. Further, rate curves seem to take on distinctly different shapes depending on the source of precursor substances. The work of Rook shows the reaction of fulvic acid solutions to be characteristic of m-dihydroxyphenyl moieties (e.g. resorcinol) as that reaction is nearly complete at near neutral pH in less than two hours (Fig. 9). Quite a different characteristic curve is observed with Ohio River water precursor and a different source of humic acid under similar conditions where the reaction takes place relatively slowly over a period of many days (Fig. 8). The probable differences in precursors at different locations has been further demonstrated in work at the EPA Cincinnati laboratory where, as expected, treatment with permanganate at low dosages was nearly 100 percent effective in preventing the formation of trihalomethanes on chlorination of resorcinol and m-dihydroxybenzoic acid solutions, yet permanganage was only marginally (10-20%) effective in reducing the ability of Ohio River precursors to form trihalomethanes upon subsequent chlorination.

### Chlorine Dose and Type

Additionally, work at the Cincinnati laboratory has shown only a slight influence on trihalomethane formation rate (or yield) occurred with increasing free chlorine dose (beyond demand) where "precursor" is kept constant (Fig. 10). Both similar (Fig. 11) and contrary results have been reported by others while working with different sources of precursor. Combined chlorine (chloramines) do not cause formation of THM's (Fig. 12).

The above serves only to indicate that although precursor materials from various supplies may be of largely natural origin, the composition of that material is likely to be different, depending on the type of supply involved and the source of precursors in the watershed. Considerably more work is needed, therefore, to understand the complex mechanisms



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TRIHALOMETHANE PRODUCTION, pH 6.7, 25°C, 10mg/I CHLORINE DOSE







Figure 10 EFFECT OF CHLORINE DOSE ON TRIHALOMETHANE FORMATION



FIG. 11 CHLOROFORM FORMATION COMPARED WITH CHLORINE RESIDUAL



of trihalomethane formation during water chlorination and to determine whether water treatment strategies for control of THM's could vary significantly among these various supplies. These various strategies are described elsewhere in these proceedings.

### FACTORS INFLUENCING THM MEASUREMENT DURING TREATMENT EVALUATIONS

### Introduction of Trihalomethane Formation Potential Concept

Because their formation is not instantaneous, THM concentrations increase in the water as it flows through a water-treatment plant (unless removed during treatment) to reach some value higher than that which would be observed if an analysis for THM species were performed immediately after sampling at the first point of chlorination. Further, the consumer is likely to receive water with THM concentrations higher than those leaving the plant because the reaction proceeds in the distribution system. This also occurs during sample storage, and in each case the concentration is time dependent. The formation rates vary according to all of the reaction conditions described in the previous section. These factors will be discussed again in the context of the THM measurement. To evaluate treatment success, four definitions are important:

1. Instantaneous THM (InstTHM) concentration-the concentration of THM in the water at the moment of sampling. This may be expressed in terms of the individual species or their sum as total trihalomethane (TTHM).

2. Terminal THM (TermTHM) concentration-the concentration of THM that occurs at the termination of the measurement of this parameter. To measure TermTHM concentration, chlorine-precursor reaction conditions are selected according to the treatment practiced at the particular plant being evaluated In general, a sample of water is chlorinated under these conditions, and chloroform and other THM species are measured after a specified time period.

TermTHM concentration is equally important as a parameter for evaluating consumer risk as is the InstTHM concentration, but because this parameter is a measure of the sum of the amounts of THM species already present (instantaneous) and those formed during the reaction time, a third parameter must be defined that is useful for evaluating unit-process performance for removal of unreacted precursor.

3. THM formation potential (THMFP)-measured as the increase in THM concentration that occurs during the storage period in the determination of the TermTHM concentration. The THMFP is obtained by subtraction of the InstTHM concentration from the TermTHM concentration either when TTHM or when the individual species data are used. THMFP is a measure of the portion of the total precursor material (of most concern to the consumer) remaining in the water at a given point in the treatment train. This parameter, when measured on unit-process influent and effluent samples, can be used to determine the efficiency of that process for removal of that pertinent fraction of precursor material.

4. Total precursor - A distinction between THMFP and a total precursor is important. Total precursor concentration is the concentration of all organic THM precursor materials present in the water that could react with halogen species under conditions that maximize the yield of trihalomethanes.

Because the identities of these organic compounds are not known precisely at this time, total THM precursor concentration also could be expressed as concentrations of THM or concentration of TTHM obtained from that reaction. No standardized procedure for measuring this parameter exists, however, and considerable research will be required to establish the optimum conditions to ensure the complete reaction of all precursors to yield maximum trihalomethane concentrations.

Because the chlorination conditions for TermTHM concentration measurement are somewhat less that optimum for THM formation, the TermTHM concentration obtained in that determination will be somewhat less than the theoretical maximum THM concentration. Thus, the value obtained for THMFP under these conditions, obtained by subtraction of the InstTHM concentration from TermTHM, will be smaller than the theoretical "total precursor" parameter. Although the value obtained (THMFP) is not the total precursor concentration, it is an index of the concentration of materials of most concern relative to THM formation at a particular water-treatment plant. Also, because controlling parameters (under treatment-plant conditions) are measured easily at the operating plant, TermTHM concentration (and, therefore, THMFP) is a practical measurement. Figure 13 presents graphically the four parameters discussed.

# Measurement of Instantaneous THM Concentrations

For InstTHM concentration measurement, the reaction of chlorine with precursor materials must be halted at the time of sampling with the goal being to measure only trihalomethanes present at the time of sampling. A small amount of reducing agent is added to the sample to react with the chlorine and, thus, render the chlorine unavailable for oxidation or substitution reactions. A small increase in trihalomethane concentrations upon storage after addition of reducing agent usually is observed; this is probably caused by a slow hydrolysis of certain trihalo-intermediates. The hydrolysis step does not require the presence of chlorine. The distinction should be made between this minor effect on the InstTHM concentration and the continued THM formation reaction when no reducing agent is added. The increase in THM concentration during storage after the addition of a reducing agent has amounted to only a few percent of the total value.

# Measurement of Terminal THM Concentration and THM Formation Potential

These two parameters are discussed together because the measurement for TermTHM concentration together with the InstTHM concentration yields the THMFP by subtraction. The TermTHM concentration is measured by reacting chlorine with THM precursors in a given sample under certain controlled conditions that affect yield and rate of formation of the trihalomethane and subsequently measuring the concentrations of THM species produced. Because



FIG. 13 FOUR TRIHALOMETHANE MEASUREMENT PARAMETERS

this is not a total-precursor-concentration measurement, the selected conditions for this measurement must be selected as appropriate according the conditions under study and be reproducible from sample to sample. As with treatment strategy selection, critical conditions to be considered are time of reaction (time elapsed before halting the halogenation reaction with a reducing agent), maintenance of a free chlorine residual, temperature, and pH.

#### Effect of Time --

Although a single measurement of THM concentrations after a storage period of several days in a bottle under appropriate conditions can give a useful determination of TermTHM concentration for that specified time, much more information can be gained from the reaction-rate curves obtained by plotting THM concentration vs time. The rate curves obtained by periodic measurement of THM concentrations of properly stored water can be used to estimate the future THM concentrations at any given time after water is taken from a sampling point within the plant, when the purpose is to use the concentration obtained to calculate the THMFP at that point in treatment for evaluation of unit-process effectiveness.

In any system, the generation of a rate curve is preferable, at least initially, so that the nature of the reaction that occurs at a location can be determined. For example, Figure 14 shows two hypothetical curves describing the rate of chloroform formation that might be expected for finished waters of distinctly different quality after leaving typical water-treatment plants.

Curves A and B in Fig. 14 represent two extreme situtations that might occur. Although at time T, the chloroform concentrations are the same for both waters, the short term chloroform concentration is greater in Plant A, and long term chloroform concentration is greater in Plant B. A Plant A curve would be expected where chloroform-formation potential is relatively low but the precursor present is of the type that reacts quickly under the given conditons i.e., the final concentration of chloroform is reached early. A Plant B curve would be expected where chloroform-formation potential is high but the reaction with chlorine is slow because of the nature of precursor or reaction conditions. Thus, these curves are more informative than a single chloroform determination performed at time T, and the single measurement at each plant easily could be misinterpreted to mean that the plant situations were the same.

Good approximations of both curves are obtained by the selection of three or four points beyond time 0 (instantaneous value) such as 1/2T, T, 2T as shown, where 2T is equal to or slightly longer than the maxiumum distribution-system residence time. These added analyses could cause the generation of rate curves to be time-consuming, especially if conditions are such that reactions are slow and the distribution-system residence time is long. If the development of the rate curve is beyond the capability of a utility, the time for the determination of TermTHM concentration should be the longest residence time in the distribution system, as this represents the most stringent conditon for that utility.



Maintenance of Chlorine Residual --

In conventional U.S. water-treatment practice, maintenance of a free chlorine residual through the distribution system often is recommended or required. As mentioned above, the continued reaction of precursor with chlorine to yield trihalomethanes depends on the maintenance of a free chlorine residual. Again, with chloroform as an example, the raw water curve presented in Figure 15 shows the abrupt cessation of chloroform production as the chlorine became depleted. The 24-hr and all later samples gave the same chloroform concentration, and chlorine-residual determinations confirmed the lack of chlorine. Thus, the 24-hr and later chloroform concentrations could be misleading, assuming one of the conditions in the water utility under investiation was maintenance of a chlorine residual throughout the distribution system. Thus, for evalaution of systems where free chlorination is practiced, to ensure that misleading results are not obtained, a chlorineresidual measurement always must be performed at the time of THM analysis to ensure that a free residual is present.

Work at the DWRD laboratory indicates that TermTHM concentrations are not affected significantly by the amount of free chlorine present. This may be only because the concentrations usually are limited by the amounts of precursors present. Because some uncertainty (see above) exists about the effect of chlorine concentration on reaction rate, the dose used in the TermTHM determination should be nearly the same as that used at the treatment plant; because that dose is adequate for maintenance of a distributionsystem residual, it should be adequate to supply the required residual for the duration of the test.

### Effect of Temperature --

Because temperature has a dramatic effect on rate of formation of THM and therefore yield at any given time, a need for close temperature control during the determmination of TermTHM concentration, therefore, is indicated. Temperature largely is controlled seasonally at a water works, and selection of a sample storage temperature will depend on the experimental objective. For example, if the objective is to estimate consumer exposure throughout a year, a logical choice is an estimated average distribution system temperature that will vary with the time of the year. When the objective is to evaluate "precursor" removal efficiencies of a unit process, the measurement should not be influenced by temperature changes and the temperature must be mainatained at a single value for all of the tests regardless of the time of the year. The investigator may even choose to run multiple tests at each sampling time including more than one storage temperature reflecting the annual range.

### Effect of pH --

The selection of the pH for the controlled reaction during the TermTHM concentration determination is less straight-forward than that for reaction time and temperature. The variation of pH through an operating water-treatment plant can be quite wide, and the variation is controlled operationally.



If the determination of the TermTHM concentration and the THMFP for the finished water only is desired, pH selection is not a problem. The samples should be stored at the finished water pH. If however, a comparison of the THMFP of the finished water with that of the source, or raw water, or with water at any stage of treatment to evaluate success of a unit process in reducing THMFP is desired, the selection of pH is more difficult.

The analyst must be sure that the same portion of the total precursor concentration (pH dependent) is reacting at each point and that the reaction rate of chlorine with that material (also pH dependent) is the same at each point. Thus, all of the samples from each of the various sampling points must be chlorinated and stored buffered at a single selected pH value. Therefore, because the THMFP test is designed to measure the portion of the total precursor that is significant in a given water as it leaves the treatment plant, the logical selection of the single reaction pH value is still that of the finished water entering the distribution system, as with the choice of temperature.

### Summary of Procedures for InstTHM, TermTHM, THMFP

Procedures for measurement of these parameters have been discussed in terms of general concepts. The specific procedures, particularly the quantatative measurement of the trihalomethanes themselves are covered in detail and a more complete way elsewhere and will not be described herein. The reader is referred to the references and EPA trihalomethane analytical procedures for detail.

In summary and review, InstTHM is the measured THM concentration when the chlorine-precursor reaction was stopped by the addition of a chemical reducing agent at the time of sampling.

TermTHM is the measured THM concentration after the reaction between precursors and chlorine has been allowed to continue in a sealed container under defined specified conditions for a given time period.

THMFP is the arithmetic difference between TermTHM and InstTHM and represents the concentration of organic precursor of concern to the analyst that is unreacted and is present in the sample at the time of the original sampling.

## Examples of the Use of Methods and Interpretation of Results

Some hypothetical examples will help to demonstrate the use of the two experimental determinations and the calculated THMFP to estimate both consumer exposure to trihalomethanes resulting from the chlorination process and the efficiences of the various unit processes within the plant for removing precursor compounds during treatment. The efficiency of unit processes for removing chloroform or other trihalomethanes can also be estimated. Simple Chlorination --

The first example, Fig. 16 represents the simplest case - a watertreatment plant with chlorination only. Figure 16 depicts the relative values for the parameters that might be obtained if analyses were conducted for the InstTHM concentration and TermTHM concentration for source water A, plant clear well, B, and a theoretical point at the maximum residence time in the distribution system, C. For simplification the triahlomethanes are being discussed here as a group. Each bar could represent the single group index (TTHM), any one of the individual species, or be subdivided horizontally into four bars of different heights to represent all four commonly found trihalomethanes.

According to the bar graph, trihalomethanes were absent in the untreated source water (InstTHM was not found on analysis of source water), but the full THMFP was present and equal to the TermTHM concentration obtained experimentally. At the clear well, some of the precursor measured as THMFP has reacted to form trihalomethanes (measured as InstTHM in finished water) leaving a smaller remaining THMFP. The remaining THMFP, plus InstTHM concentration equals the TermTHM concentration determined originally on the source water. At point C the entire original THMFP had reacted to give an InstTHM concentration identical to the TermTHM concentration.

No unit process exists at this plant that was effective for reduction of either TermTHM or InstTHM concentrations. The practice of chlorination itself converted THMFP to InstTHM, thereby causing a reduction in the THMFP. In assessing the THMFP removal by any unit process, care must be taken to separate removal of THMFP by conversion to InstTHM by chlorination, and removal of THMFP by the unit process itself. Only at a point closer to the treatment plant than the maximum length in the distribution system is consumer exposure to THM lower than the TermTHM concentration shown in Fig. 16.

Conventional Treatment ---

Shown in Fig. 17, during conventional treatment with raw water chlorination, some THM is formed during rapid mixing and throughout the following treatment stages in the presence of chlorine. Thus, the InstTHM concentration increases as the water passes through rapid mixing, settling, and filtration, points B, C, and D. Coagulation and settling do reduce THMFP (i.e., precursor removal) so that parameter as well as TermTHM concentration declines from point B to C. Filtration removes a little more precursor material that is associated with the carryover floc; therefore, the THMFP declines slightly again from point C to D. The remaining THMFP is converted by chlorination to THM from point D to E, and therefore, the InstTHM concentration determined for a sample taken at that point in distribution equals the TermTHM concentration of the sample from point D.



TREATMENT BY CHLORINATION ONLY



# FIG. 17 TRIHALOMETHANES FORMED DURING CONVENTIONAL TREATMENT WITH RAW WATER CHLORINATION

Summary of Examples --

These two hypothetical examples should not be considered to be predictions of the success or failure of certain unit processes in a treatment train or to be indications of the relative effectiveness of those processes. The examples do serve to indicate the kinds of results that might be obtained when a plant is sampled for measurement of InstTHM and TermTHM concentrations and THMFP and when the results of these measurements are compiled for unit processes or whole plant evaluations.

### Summary of Formation Potential Concept

Instantaneous trihalomethane concentrations in chlorinated drinking water may be measured in samples where chlorination reactions were stopped by addition of a suitable reducing agent at the time of sampling. The trihalomethanes then are separated from the aqueous phase and subjected to an acceptable form of measurement.

Terminal trihalomethane concentration is a measure of trihalomethanes formed as a result of sample storage under conditions that closely approximate those of the distribution system corresponding to the plant under study. The parameter can be used to estimate consumer exposure to trihalomethanes as well as provide a route to the calculation of trihalomethaneformation potential remaining at any stage of treatment.

Trihalomethane-formation potential is a useful measure of pertinenent unreacted precursor material. The value is obtained by subtraction of the instantaneous trihalomethane concentration from the terminal trihalomethane concentration in a given sample.

Total precursor concentration measured as maximum trihalomethane produced on chlorination is not a viable parameter because establishing completeness of the reaction is rather difficult and the measurement invariably would give trihalomethane concentrations higher than those actually reaching the consumer.

Generation of the trihalomethane-formation-rate curve, although not always necessary, provides useful background information for plant and unitprocess evaluations. The curve, when generated for finished water samples, provides a useful estimate of trihalomethane concentrations for any given time after the water leaves the treatment plant.

The proper measurements of instantaneous trihalomethane and terminal trihalomethane concentrations and calculation of trihalomethane-formation potential in conjunction with a carefully planned sampling program can be used to determine success or failure of efforts to reduce trihalomethane concentrations reaching the consumer in modification of water-treatment practice.

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