

Chlorination of HPC washed from water mains

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ABSTRACT

The installation of new mains into water distribution systems, or the repair of existing mains following breaks, often necessitates the disinfection of the system which had been repaired. This is generally accomplished (at least in the US) by chlorination. The basis for existing criteria for the disinfection in such cases has not been placed on a firm technical footing. Furthermore, pressures for cost reduction (and thus reduction in chemical use) are providing incentives for reexamination of current practice. In this work, we present results of studies on the inactivation of heterotrophic plate count organisms washed from actual pipe sections by chlorine under conditions used for main disinfection. Sodium and calcium hypochlorite, as well as dissolved chlorine, are used as germicidal agents. It is concluded that current practices can achieve high levels of reduction of these indigenous organisms.

Key words | chlorination, disinfection, distribution system, mains repair, plate count

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INTRODUCTION

Inadequate care during repairs to distribution systems has been the cause of a number of waterborne disease outbreaks associated with public water systems (Herwaldt *et al.* 1991). Disease outbreaks may result from gross contamination during breaks or repairs in mains followed by insufficient disinfection. Also, some water distribution systems may harbor particulate material arising from pre-installation conditions, such as dust, soil particles or animals which were in the mains prior to installation, or they may develop their own microbial flora from organisms introduced via new mains or during repairs. Such exogenous bacteria may become established within a distribution system by regrowth, perhaps as a biofilm, and may thereby become protected from the action of disinfectants (Boardman & Sproul 1977; LeChevallier *et al.* 1981, 1990; Haas *et al.* 1983a, 1983b; Stewart & Olson 1992a, 1992b). Therefore, disinfection of mains after repair, or installation of new pipe, is an important public health protection measure.

Conventional practices for water mains disinfection in the United States are summarized in the American Water

Works Association *Standard C651* (AWWA 1993). The standard covers newly installed mains ready to be put into service, mains ready to be put back into service after a break has been repaired, and mains which continue to yield water samples with coliform or other indicator bacteria. The standard describes three methods for disinfection:

- The first is a tablet method that is considered to be suitable for new pipes that have been kept clean. Calcium hypochlorite tablets are used to achieve a concentration of 25 mg/l of free chlorine. A holding time of 24 hours is recommended for temperatures above 5°C. There is no specific recommendation for the chlorine residual at the end of the holding period, therefore there is no specific 'CT' (product of disinfectant concentration and time) requirement.
- The second method requires flushing of a new main with strong calcium hypochlorite solution (up to 100 mg/l), followed by filling the main with water with at least 25 mg/l free chlorine. A final residual of

at least 10 mg/l free chlorine after 24 hours should be achieved.

- The third method uses a slug dose of free chlorine (concentration >100 mg/l) in contact with the pipe surfaces for at least 3 hours with a chlorine residual in excess of 50 mg/l (measured at the end of the pipe section). An extreme version of this procedure (section 10.4 of the standard) is used for main break situations and/or 'situations with dosing up to . . .' 300 mg/l of chlorine for as little as 15 minutes with a 50 mg/l residual.

Various water utilities in the US have adopted the AWWA standard, or modified forms of it, to use as specifications in contracts for installation of new pipeline or repair of existing pipes. However, there has been little specific technical justification for the adequacy of the existing standards. Buelow *et al.* (1976) provided data on the inactivation of standard plate count organisms by chlorine during pipe disinfection, and showed that a dose of 25 mg/l would result in 4 logs reduction of SPC (35°C incubation of pour plates for 48 hours) organisms after 1 day of exposure. However the details of their analysis were not completely specified. The objective of this work was to provide data on the sensitivity of microorganisms washed from pipes to disinfection conditions specified in the AWWA standard.

MATERIALS AND METHODS

New and used ductile iron (lined and unlined) main sections were obtained from the City of Philadelphia Water Department. PVC main samples were obtained from J. M. Manufacturing, New Brunswick, New Jersey. One used section of asbestos cement main was obtained from the Philadelphia Suburban Water Company. Upon receipt, the ends of the pipes were sealed with sterile kraft paper and the pipes were stored in dry ambient temperature conditions until use.

Bacteria associated with new pipe, as measured by the HPC method, were obtained by washing full sections of pipe. Three sequential batch washes with dechlorinated

Philadelphia tap water were made, and combined to obtain the HPC organisms. These were performed by flowing the water through the pipe at approximately 0.8 m/s, and recirculating for 30 minutes. The detailed washing procedure has been previously described (Haas *et al.* 1999). The initial HPC levels ranged from 500–14,800 per ml, with a geometric mean of 2060/ml.

Disinfection kinetics experiments

Kinetic experiments were conducted using the flush water to study the sensitivity of water-main associated organisms to disinfection. A total of 33 experiments were conducted as shown in Table 1.

Stock chlorine solutions

Three forms of chlorine were used as disinfectants. The stock solutions were prepared as follows:

- *Calcium hypochlorite solution.* The working stock solution was prepared to a concentration equal to 2000 ± 100 mg/l by dissolving calcium hypochlorite granules in deionized water.
- *Sodium hypochlorite solution.* The working stock solution was prepared to a concentration equal to 4000 ± 100 mg/l by diluting strong sodium hypochlorite (e.g. 5%) solution with deionized water.
- *Hypochlorous acid solution.* The working stock solution was prepared to a concentration equal to 2000 ± 100 mg/l by bubbling chlorine gas into deionized water.

Analysis for residual

Chlorine levels were determined by the DPD colorimetric methods using a digital titrator (Hach Company, Loveland, Colorado).

Equipment setup

Ten 2-l heat-resistant glass beakers were used as reaction vessels. Samples were collected from the reactor vessels by

Table 1 | Summary of experiments performed

Pipe material	Diameter (inches)	Length	Number of experiments
New pipe			
PVC	2 (51 mm)	10 ft (3 m)	2
	4 (102 mm)	10 ft (3 m)	2
	8 (203 mm)	10 ft (3 m)	2
Ductile iron with asphalt lining	6 (152 mm)	18 ft (5.5 m)	3
	8 (203 mm)	18 ft (5.5 m)	3
Ductile iron without lining	3 (76 mm)	20 ft (6.1 m)	3
	4 (102 mm)	18 ft (5.5 m)	3
Used pipe			
Ductile iron	6 (152 mm)	9–27 in (23–69 cm)	9
	8 (203 mm)	10–22 in (25–56 cm)	6
	12 (305 mm)	23 in (58 cm)	1
Cast iron	6 (152 mm)	28 in (58 cm)	1
Asbestos cement	8 (203 mm)	28 in (58 cm)	1

using presterilized high-density polyethylene (HDPE) syringes (catheter tip syringe, no. 9664, Becton-Dickinson, Rutherford, NJ). Each reactor vessel was sufficiently mixed by a submersible magnetic stirrer (model 700, Troemner, Inc., Philadelphia, PA) or a multistirrer (MC 303, Scinics Co., Ltd, Tokyo, Japan) and a larger star-shaped stir bar (star head stir bar, 58 × 15 mm; Fisher Scientific, Pittsburgh, PA). The temperature of the reactor vessels was held constant at 15°C using a circulating refrigerated water bath (Techne, no. RB-12, Techne, Inc., Princeton, NJ).

Performance of the experiment

Ten reactor vessels were run in parallel (see Figure 1). One reactor was used as control to check for the presence of

microbial decay/growth in the absence of disinfectant. The other 9 reactors were used for survival and residual measurement when subjected to three different forms of chlorine at three dose levels. The following disinfectant doses were employed:

- Experimental water (control)
- Experimental water + Calcium hypochlorite 100 mg/l
- Experimental water + Calcium hypochlorite 50 mg/l
- Experimental water + Calcium hypochlorite 25 mg/l
- Experimental water + Sodium hypochlorite 100 mg/l
- Experimental water + Sodium hypochlorite 50 mg/l
- Experimental water + Sodium hypochlorite 25 mg/l
- Experimental water + Hypochlorous acid 100 mg/l
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- Experimental water + Hypochlorous acid 25 mg/l

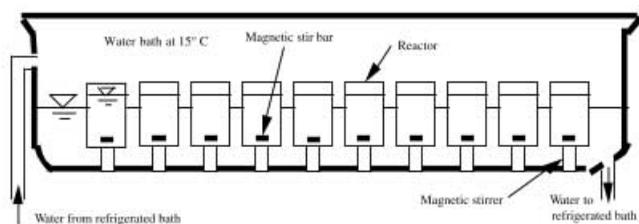


Figure 1 | Schematic of experimental layout.

To initiate the experiment, each reactor vessel was filled with 1900 ml of wash water from the first flush of the mains. Mixing was initiated. At zero time (start of the experiment), a solution of disinfectant was added to all the reactors except the control reactor. Different volumes of stock solutions were diluted to 100 ml, and added to the reactors to provide 25, 50 and 100 mg/l of chlorine doses (three reactors for each form of chlorine). The control reactor received the same volume of flush water without the addition of disinfectant.

At predetermined times, including zero time, samples of approximately 50 ml were taken from the control and survival reactors. Additional samples were collected for microbial enumeration at 5 seconds, 15 minutes, 1 hour, 6 hours, 12 hours, and 24 hours. Disinfectant residual was measured at zero, 6, and 24 hour times. Samples collected for microbial enumeration were immediately dechlorinated with a stoichiometric excess of sterile sodium thiosulfate.

Heterotrophic plate count enumeration

Microbial samples collected for enumeration were decimally diluted as necessary to achieve a countable number of colonies per plate. For preparing decimal dilutions of organisms prior to enumeration, phosphate buffered dilution water was prepared and autoclaved at 121°C for 15 minutes (APHA *et al.* 1989: *Standard Methods*, Section 9050 C., p. 9–31).

Samples were suspended in dilution water for no more than 30 minutes at room temperature to avoid death or multiplication of microorganisms (APHA *et al.* 1989: *Standard Methods*, p. 9–31).

The diluted samples were membrane filtered through 0.45 µm membrane filters. The filters were placed on sterile agar plates with R2A agar. R2A agar was used according to the manufacturer's specifications. The medium was sterilized by autoclaving after preparation (APHA *et al.* 1989: *Standard Methods*, Section 9215 A).

All the colonies on R2A medium after incubation at 25°C for 5 days were counted to give the heterotrophic plate count.

Kinetic analysis of inactivation

Disinfection residual decay analysis

The following analysis was based on the chlorine residual measurements taken from all nine reactors for all experiments. Residuals were measured at 0, 6 and 24 hours. Decreases in residuals are attributed to demand caused by organisms and materials flushed from the mains, and to volatilization. Higher chlorine decay was observed in reactors with sodium hypochlorite and calcium hypochlorite than those with HOCl.

The residual data were analyzed for the value of k^* (constant) according to the first-order decay rate equation:

$$C = C_0 \exp(-k^*t) \quad (1)$$

Where C = observed disinfectant residual (mg/l)

C_0 = initial disinfectant dose (mg/l)

t = time from start of experiment to time of sample

The best fit values of k^* were determined by non-linear regression in which the sum of squares, $([C_{\text{predicted}} - C_{\text{observed}}]^2)$, was minimized. This was done using the Solver add-in function with Microsoft® Excel®. These k^* values were later used in the Chick-Watson and Hom models (described in the following section) to fit the survival data.

Kinetic models with first order decay of residuals

The Hom model (Hom 1972) was used to fit the inactivation data obtained from the kinetic experiments conducted with wash water from the water mains. This was

modified to account for first order decay (using the approximation derived in Haas & Joffe 1998).

Nonlinear regression was used to fit Chick-Watson and Hom models with first order decay of residuals. The best-fit parameters k , n , and m were estimated as the values that resulted in the error sum of the squares, ESS being the minimum.

$$ESS = \sum (\ln Si - \ln Si^*)^2$$

Where Si^* = predicted survival ratio

Si = observed survival ratio

A Matlab® program was written for optimization (minimizing error sum of squares).

RESULTS & DISCUSSION

Figure 2 provides an example of results from a single experiment—in this case the disinfection of HPC organisms obtained from an 8-inch (203 mm) diameter new PVC pipe section. Typically, for a clean pipe, the decay of disinfectant was less than 10% of the applied residual at the end of 24 hours of contact. The fitted lines represent the best-fit Hom kinetic models applied to these data. In all cases, the Hom model provided a statistically significant improvement in fit versus the Chick-Watson model. Qualitatively, this is shown by the evident ‘tailing’ in the inactivation curves. For all experiments, the extent of inactivation by hypochlorous acid was greater than for the other two disinfectants. This is readily explained by a pH effect—the pH after HOCl addition was below (often by as much as 3 pH units) the pH after addition of either NaOCl or $\text{Ca}(\text{OCl})_2$.

Hom model parameters were determined for each of the 36 experiments, the three different disinfectants being considered separately. Thus a total of 108 sets of Hom model parameters (k , n , m) were estimated. These parameters relate the survival ratio of organisms to the concentration of the disinfectants and the contact time. This efficacy of the disinfectants can be shown by means of C-T (chlorine residual concentration \times contact time) plots. Note that time is plotted in hours rather than in minutes (as is common practice for water disinfection *per se*). The

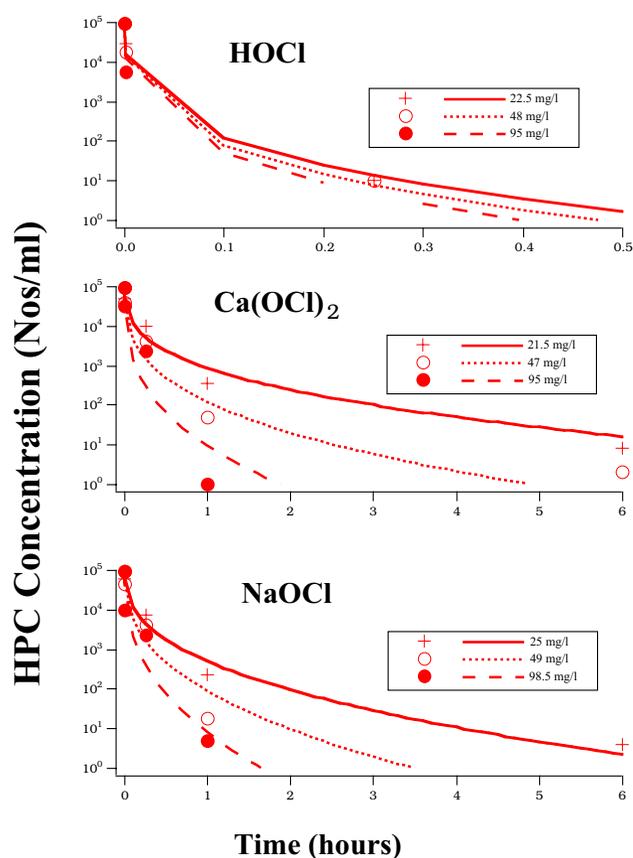


Figure 2 | Disinfection of bacteria obtained from an 8-inch (203 mm) PVC pipe section (15°C).

different concentrations of disinfectants that would effect a constant level of ‘kill’ are plotted against the time values required to produce that ‘kill’ (assuming for illustration no decay of disinfectant). A value of 99.99% removal of organisms was chosen for comparative purposes as this represents a substantial level of disinfection. These C-T plots (with the three different disinfectants) are shown in Figures 3 to 5. In these figures, it is assumed (for comparative purposes) that there is no loss of chlorine residual. In the same figures, the C-T point corresponding to the tablet method (25 mg/l for 24 hours) is also shown. It can be observed from these figures that the C-T required for 4 log removal was always less than the C-T from the tablet method. However, when developing these C-T plots the chlorine demand from the mains was not taken into consideration, which means that the actual C-T required

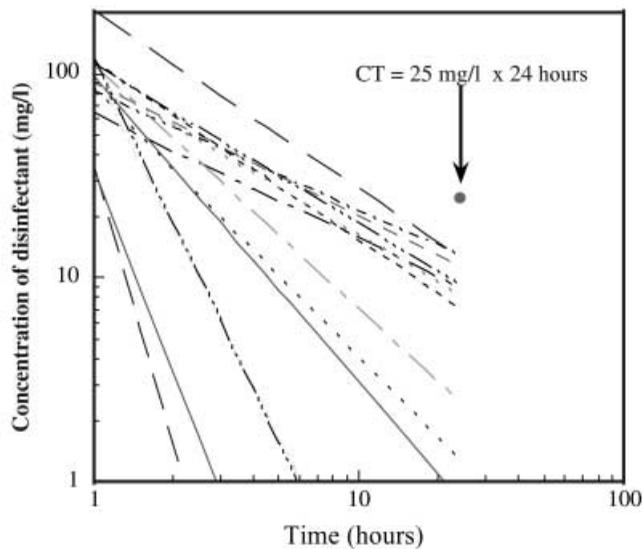


Figure 3 | CT lines for 4 log inactivation with $\text{Ca}(\text{OCl})_2$ under demand free conditions at 15°C compared to CT under standard practice.

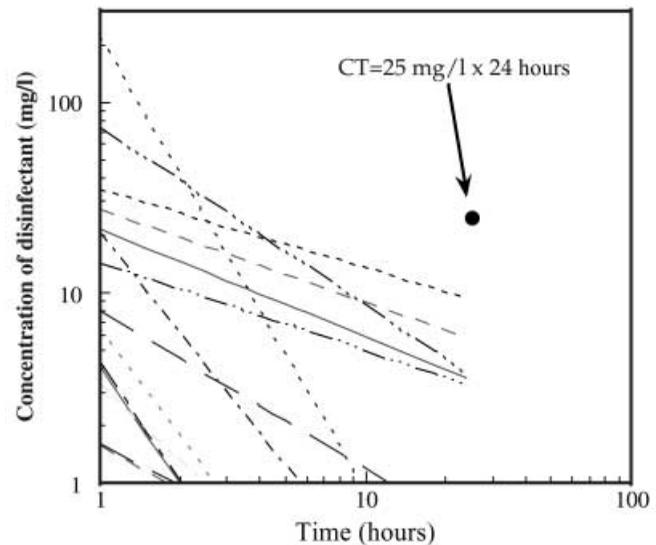


Figure 5 | CT lines for 4 log inactivation with HOCl under demand free conditions at 15°C compared to CT under standard practice.

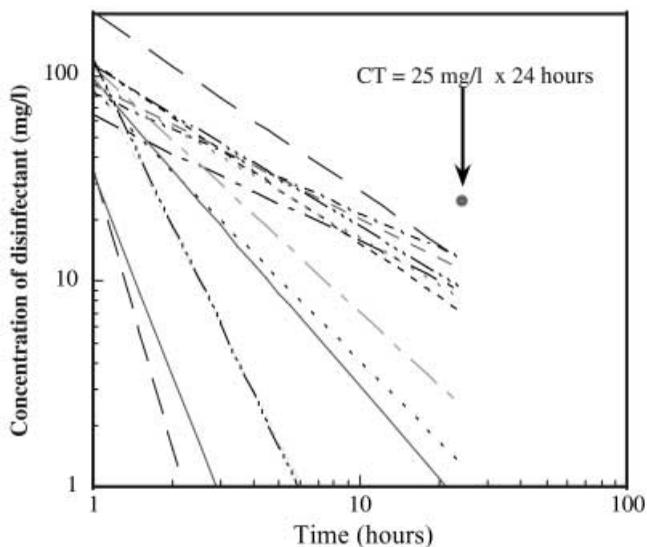


Figure 4 | CT lines for 4 log inactivation with NaOCl under demand free conditions at 15°C compared to CT under standard practice.

based on initial applied dose will be higher than the C-T from these figures.

If chlorine doses (and thereby residuals) could be reduced while maintaining sufficient disinfection of mains, as perhaps suggested by these plots, then reduced potential discharge of chlorinated water (or reduction

of the cost of dechlorination) could be achieved. A survey of water utilities in the US indicated that nearly 12% of dechlorinated water comes from disinfection of mains, prior to discharge or disposal (Haas *et al.* 1998).

The data presented graphically in Figures 3 to 5 show that there was considerable variation among the specific inactivation parameters from run to run. An evaluation was made to ascertain whether this variation was statistically significant, or whether it could (to an equivalent degree) be described by a smaller set of parameters dependent (e.g.) upon chemical applied, pipe material or pipe condition. The entire data set was subdivided and the best set of Hom parameters corresponding to each subset was obtained. The overall sum of squares (by summing over each subset) characterizes the goodness of fit. The partitioning into subsets was performed using several discriminators, as follows:

- Use a single set of parameters (k, m, n) to depict the entire data set.
- Use a set of parameters for each disinfectant (NaOCl, $\text{Ca}(\text{OCl})_2$, HOCl).
- Use a set of parameters for each pipe diameter.

Table 2 | Comparison of fit using different partitions of the data

Partitioning	SS	Parameters	df	MS	sd	p (vs individual experiments)
Each experiment (35) treated separately for each disinfectant	998.38	105	607	1.645	1.282	
2 disinfectant classes × 5 materials (10 combinations)	1100.02	30	682	1.613	1.270	8.52E-01
Ca(OCl) ₂ vs NaOCl vs HOCl	1435.32	9	703	2.042	1.429	7.02E-14
(CaOCl/NaOCl) vs HOCl	1438.33	6	706	2.037	1.427	1.57E-13
Pipe material	1569.10	15	697	2.251	1.500	2.23E-23
Age and tuberculation	1650.67	9	703	2.348	1.532	4.74E-27
Pipe diameter	1638.40	18	694	2.361	1.536	1.81E-28
Single pooled model	1849.61	3	709	2.609	1.615	2.11E-37

NOTES: SS is the sum of squares of differences between fitted and observed natural log survival ratios.

df is degrees of freedom (observations minus number of parameters).

MS (mean square) equals SS divided by df.

sd (standard deviation) is the square root of MS.

p is the significance level of the difference between the partition and the fit in which each experiment and disinfectant is treated as a special case (a p value below 0.05 indicates a statistically significant difference).

Parameters refer to the kinetic model 'constants' obtained by fitting the data.

- Use a set of parameters for each degree of use of the pipe. For this comparison pipes were classified as new versus used. Used pipes were sub-classified based on visual observation as clean surfaces versus tuberculated surfaces.
- Use a set of parameters for each type of pipe material (ductile iron lined, ductile iron unlined, PVC, asbestos cement, cast iron).

A summary of the analysis is shown in Table 2. In this table, the statistical significance of the fit is assessed by comparison with the fit in which each experiment–disinfectant combination is fitted separately. In other words, is the deterioration (in sum of squares) significantly different from the separate experiment–disinfectant fit when the reduction in number of fitted model parameters is taken into account? This comparison was performed using an F test.

Using the five partitions enumerated above, it was found that, while each partitioning gave an improvement

in fit relative to using only a single set of parameters, these did not capture the variability demonstrated by analyzing each experiment–disinfectant separately. It was also noted, in examining the results by partitioning by disinfectant, that the inactivation using sodium hypochlorite and calcium hypochlorite was virtually identical. This is also shown by a comparison of survival results for data obtained in the same experiment, and at the same contact time, but differing only in the use of sodium versus calcium hypochlorite (Figure 6). Hence an additional partitioning of data was conducted in which the sodium and calcium hypochlorite data were pooled, and the second subset was the hypochlorous acid data. It is seen that the sum of squares for this two-segment partitioning was virtually identical to that considering the three disinfectants separately.

The comparative behavior of the disinfectants is probably due to the effect of the agents on pH. Figure 7 summarizes the mean and standard deviation of water pH as a function of disinfectant and chemical dose.

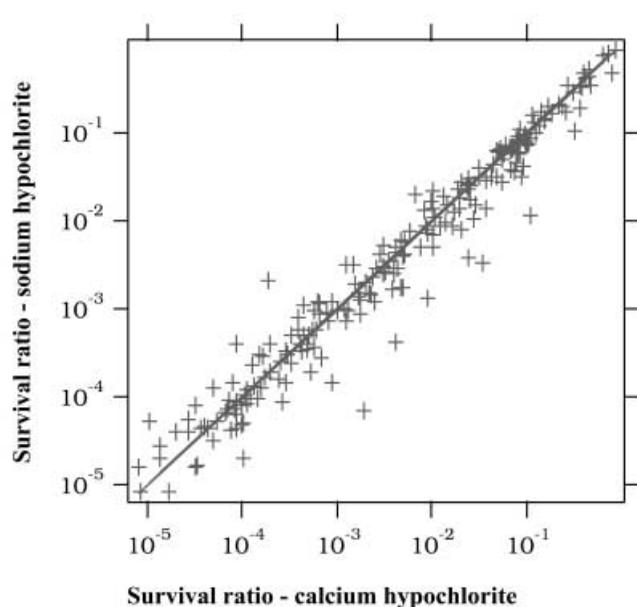


Figure 6 | Comparison of survival ratios—sodium versus calcium hypochlorite.

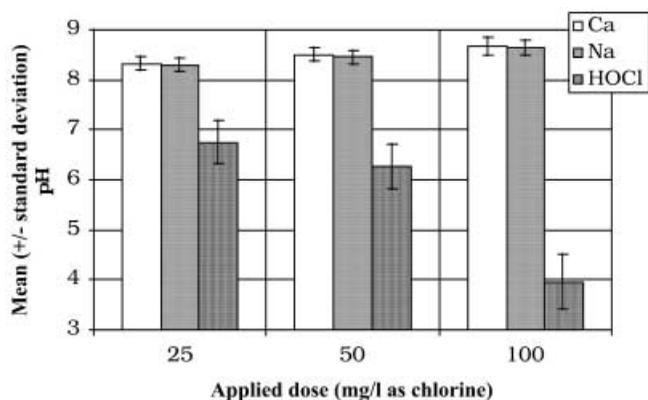


Figure 7 | Water pH versus dose and disinfectant. Mean \pm standard deviation of all experiments.

A three-way analysis of variance (time \times agent \times concentration) showed no statistically significant difference due to time, while both agent and concentration showed a statistically significant effect on pH. A further (partial-F) test showed that any difference in pH resulting from calcium and sodium hypochlorite was not statistically significant, while the pH from HOCl was different from the other two agents. It should be noted that the water used in these experiments was relatively poorly

buffered (alkalinity ranging between 80 and 90 mg/l as CaCO_3) and had an initial pH averaging 8.15. In a more strongly buffered water, the difference between pH values obtained from the three agents would probably be smaller.

It was decided to look at models in which two attributes were used to partition the data, so as to obtain a set of kinetic parameters over very few conditions which could adequately describe the variability in disinfection performance. Thus, a partitioning with respect to both disinfectant (in two classes—HOCl versus the sodium/calcium hypochlorites) and pipe material was conducted. This produced 10 different subsets of data. As shown in Table 2, this approach provided a fit that was indistinguishable from one using experiment–disinfectant as the discriminators.

The best-fit kinetic parameters for the model involving discrimination on the basis of pipe material and disinfectant are shown in Table 3. For HOCl with cast iron and asbestos-cement, these constants may contain substantially greater uncertainty due to the relative paucity of data points for these subsets. In Figure 8, the 'CT' lines for 4 logs inactivation (under demand-free conditions) are plotted. Except in the case of HOCl with cast-iron (where the small number of data points limit the precision of estimation), all of the subset lines are less than the AWWA standard that specifies a 25 mg/l residual after 24 hours.

It should be noted that the summary in Figure 8 and the statistical analysis of inactivation is performed after correction for chlorine demand. Hence, while there is some effect of pipe material, the magnitude of this effect is relatively small. This indicates that the microorganisms derived from the different types of pipe (and the chemical compositions resulting from the interaction of water with the pipe surface) do not differ substantially. However, in the actual disinfection of pipe *in situ*, there may be more substantial differences due to different rates and amounts of chlorine demand exerted by different surfaces. This latter effect is discussed in our full report (Haas *et al.* 1998), and will be the subject of a further paper in preparation.

The standard deviation of estimation from the partitioning based on pipe material and disinfectant type is 1.2 (Table 2) natural log units. This is equivalent to a standard deviation of 0.5 \log_{10} units. Based on prior

Table 3 | Hom inactivation parameters for data partitioned by pipe material and disinfectant

Disinfectant	Material	<i>k</i>	<i>m</i>	<i>n</i>	Number of observations	SS
Calcium or sodium hypochlorite	Lined ductile iron	4.5808	0.1637	0.1390	387	491.23
Calcium or sodium hypochlorite	Unlined ductile iron	4.1645	0.1861	0.1777	119	187.95
Calcium or sodium hypochlorite	PVC	1.8459	0.3124	0.3417	120	142.90
Calcium or sodium hypochlorite	Asbestos-cement	5.4043	0.2819	0.0986	20	0.79
Calcium or sodium hypochlorite	Cast iron	6.0250	0.1588	0.1087	15	11.75
HOCl	Lined ductile iron	5.7949	0.1734	0.1603	123	168.77
HOCl	Unlined ductile iron	7.3068	0.1395	0.0721	40	64.28
HOCl	PVC	5.7771	0.2933	0.2318	29	32.31
HOCl	Asbestos-cement	4.1952	0.2109	0.1869	5	0.001
HOCl	Cast iron	1.8238	0.1283	0.3749	4	0.03

disinfection studies with diverse organisms, this appears to be consistent with the intrinsic level of variability which such work contains (Haas *et al.* 1995).

The utility of the kinetic models developed herein must be taken in context with the chlorine demand exerted during disinfection by the liquid in the pipe, and the pipe wall. The latter in most cases is the dominant source of demand. Given this information, the appropriate initial dose of disinfectant necessary to achieve a particular log-reduction in HPC levels may be computed. Alternatively, given information on the initial HPC load in the pipe (Haas *et al.* 1999), and the final desired (after disinfection) HPC level, the necessary dose and contact time can be computed. As an example, the goal of the Philadelphia Water Department is to achieve an HPC level below 100/ml after main disinfection.

CONCLUSIONS

This work has shown that the Hom model (taking disinfectant demand into account) provides an adequate fit to

the kinetics of inactivation of HPC organisms (derived from pipe surfaces) by chlorine under conditions used for pipe disinfection. The kinetic parameters vary as a function of disinfectant type (sodium or calcium hypochlorite versus HOCl), and pipe material.

Among the three different forms of chlorine studied, hypochlorous acid was much more effective than sodium and calcium hypochlorites. This could be because of the lower pH in the hypochlorous acid reactors. Both the hypochlorites provided similar inactivation indicating that these can be used interchangeably for a specific inactivation requirement depending on the field conditions. It should be noted that this study did not consider possible limitations from the rate of dissolution of calcium hypochlorite tablets, which might result in the practical need for somewhat greater contact time (versus sodium hypochlorite) when they are applied.

In all cases, regardless of material, diameter, or age of pipe, the combination of 24 hours at a 25 mg/l residual resulted in more than four logs inactivation of HPC organisms. Based on this, the current AWWA standards (based on the 24 hour \times 25 mg/l combination) are more than

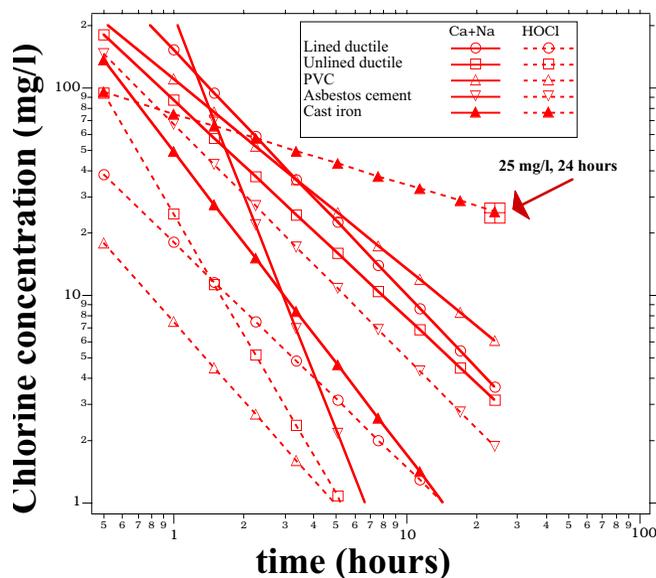


Figure 8 | CT lines for 99.99% inactivation of HPC organisms using parameters partitioned by pipe material and disinfectant.

adequate, and further study to assess the potential for chlorine reduction should be conducted.

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