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Factors affecting the formation of trihalomethanes in the presence of bromide during chloramination*

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Abstract: The effects of the concentration of dissolved total organic carbon (TOC), the TOC/Br^- ratio, bromide ion levels, the chlorine to ammonia-N ratio (Cl:N), the monochloramine dose and the chlorine dose on the formation of trihalomethanes (THMs) (including chloroform, bromodichloromethane, chlorodibromomethane, and bromoform) from chlorination were investigated using aqueous humic acid (HA) solutions. The profile of the chloramine decay was also studied under various bromide ion concentrations. Monochloramine decayed in the presence of organic material and bromide ions. The percentage of chloroform and brominated THMs varied according to the TOC/Br^- ratio. Total THMs (TTHMs) formation increased from 112 to 190 μg/L with the increase concentrations of bromide ions from 0.67 to 6.72 mg/L, but the chlorine-substituted THMs were replaced by bromine-substituted THMs. A strong linear correlation was obtained between the monochloramine dose and the formation of THMs for Cl:N ratios of 3:1 and 5:1. These ratios had a distinct effect on the formation of chloroform but had little impact on the formation of bromodichloromethane or chlorodibromomethane. The presence of bromide ions increased the rate of monochloramine decay.

Key words: Trihalomethanes (THMs), Humic acid (HA), Bromide, Monochloramine
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1 Introduction

More than 500 disinfection by-products (DBPs) have been detected in drinking water (Clark, 1998). Trihalomethanes (THMs) and haloacetic acids (HAAs) are two primary species of DBPs that are found in water disinfected with chlorine or chloramines. These DBPs constitute a health issue, since they are toxic, carcinogenic, and mutagenic to humans, or induce reproductive and developmental problems (Glaze *et al*., 1993; Minear and Amy, 1996; Simmons *et al*., 2002; Oliveira *et al*., 2006). Bromide, which occurs in natural waters, can be oxidized immediately by chlorine to the active oxidant hypobromous acid (HOBr), which can react with natural organic matter (NOM) to form brominated DBPs. Some bromide-containing DBPs pose more severe health threats than their chlorinated analogs (Bull and Kopfler, 1991).

Chloramines are often used by many utilities as a secondary disinfectant in the distribution system because they are less reactive than free chlorine and produce lower levels of DBPs. New regulations on THMs and HAAs have also forced many drinking water utilities to switch to chloramination (USEPA, 1998).

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NOM includes humic and fulvic acids in heterogeneous mixtures. Some components of NOM, namely phenols, amino acids, and dihydroxybenzene compounds, have been recognized as DBP precursors (Minear and Amy, 1996; Rostad *et al*., 2000; Gallard and von Gunten, 2002). Various NOM compositions generate a variety of mixtures of DBPs and may show different reactivities with chlorine or bromine.

Factors, such as the concentration of total organic carbon (TOC), pH, temperature, disinfectant dose, reaction time, and precursor characteristics, can affect the production of DBPs (Platikanov *et al*., 2010; Wang *et al*., 2010). When bromide ions occur in raw or finished water, their presence and concentration can affect the chlorination DBPs because bromine shows a higher reactivity with NOM compared to chlorine. Many studies have found that bromide contributes to THMs and HAAs when the levels of chlorine and bromine are similar. Bromide ions can react with chlorine to produce HOBr, which is a more efficient substituting agent than HOCl, while HOCl is a better oxidant than HOBr. The result of a kinetic analysis indicated that the reaction rate of HOBr with NOM is higher than that of HOCl by a factor of about ten (Westerhoff *et al*., 2004).

The objective of this study was to investigate the important factors that affect the formation of four THMs during chloramine disinfection in a prototype laboratory simulation. The reaction of NOM with chlorine and bromine is the key issue in controlling DBPs in drinking water treatment processes and in the distribution system. Four THMs ($CHCl_3$, $CHCl_2Br$, $CHClBr_2$, and $CHBr_3$) were chosen for analysis in this study.

2 Materials and methods

2.1 Experimental design

The experiments were designed to investigate the effect of TOC on the formation of THMs. Working solutions of HA (TOC=2.0, 4.9, 9.5 mg/L) were prepared and adjusted to pH 7.5 with 1 mmol/L phosphate buffer. A monochloramine dose of 1 mg/L (Cl:N=3:1, weight ratio) and a bromide ion dose of 3 mg/L were added to each of the HA working solutions. Additional experiments were carried out to study the impact of the TOC/Br^- ratio on the THMs species. A chlorine dose of 2 mg/L and bromide ion doses of 0.67, 2.02, 3.36, 4.71, and 6.72 mg/L were spiked into the HA working solutions at three TOC levels (2.0, 4.9, and 9.5 mg/L).

Another experiment was conducted to investigate the effect of the bromide ion level on THMs formation and the distribution of the four THMs. The selected TOC dose was 5 mg/L for the HA solution. Prior to chloramination, a series of HA solutions were spiked with 0.67, 2.02, 3.36, 4.71, or 6.72 mg/L of bromide, respectively. A chlorine dose of 2.0 mg/L was added to an HA solution adjusted to pH 7.5 with HCl and/or NaOH.

Monochloramine (5 mg/L) (at a Cl:N ratio of 3:1 or 5:1) was added to compare THMs formation with the distribution of the four THMs.

After a reaction time of 24 h, the residual chloramines were quenched in dark with a stoichiometric amount of 10% sodium thiosulfate at 25 °C. The solution was then extracted with pentane for immediate analysis of the THMs by gas chromatography with electron capture detection (GC/ECD) (Model GC-2010, Shimadzu, Japan).

2.2 Chemicals and materials

The precursor material was prepared by dissolving 1 g of commercial HA (Sigma-Aldrich Pte. Ltd., USA) in ultrapure water. The pH of the stock HA solution was adjusted to 10 by adding 1 mol/L NaOH. The solution was then filtered through a 0.45-μm membrane (Millipore Corp., USA) and stored at 4 °C in a refrigerator. The stock HA solution was diluted with ultrapure water to obtain the target TOC concentration. All glassware was washed with dilute nitric acid and rinsed three times with tap water and ultrapure water, consecutively. All cleaned glassware was baked for 12 h at 180 °C in a drying oven to remove volatile organic compounds and to provide chlorine demand-free glassware. Disinfectant solutions were made up with ultrapure water. Stock solutions of 400 mg/L sodium hypochlorite and 25 g/L ammonium chloride (Sinopharm Chemical Reagent Co., Ltd., China) were prereacted to form Cl:N 5:1 and 3:1 monochloramine solutions.

2.3 Analytical methods

The TOC (after pre-filtration through a 0.45-μm membrane) was analyzed using a total organic carbon analyzer (TOC-VCPH, Shimadzu Co., Japan).

Monochloramine analysis was performed using a pocket colorimeter (Cat. No. 46700-00, Hach Company, USA), as described by the Standard Method (APHA, 1998).

The four chlorine and bromine containing THMs were quantified by liquid/liquid extraction with pentane and by GC/ECD based on the Standard Method (APHA, 1998). The GC temperature program includes an initial temperature of 40 °C for 2 min, ramping to 100 °C at 10 °C/min and to 240 °C at 20 °C/min and maintaining 240 °C for 1 min. The relative standard deviation of the standard THMs solutions was within 3.8%–5.1%.

3 Results and discussion

3.1 Effect of the TOC concentration and the TOC/Br$^-$ ratio on the formation of THMs

The TOC level has been used as an indicator of the presence of organic matter in drinking water. The fulvic acid and HA components of the TOC are important precursors for THMs (Christman *et al.*, 1990). Based on bench-scale and field experiments, many investigators have found that the higher the TOC value, the higher the levels of THMs formation (Watson, 1993; Diehl *et al.*, 2000; Rodrigues *et al.*, 2006). Fig. 1 shows the impact of the TOC level on the yields of THMs. Increasing the TOC levels resulted in substantially increased total THMs (TTHMs) production. The reason for this is that the formation originates from HA (Rodrigues *et al.*, 2006), and at higher TOC levels there are more reactive organic sites. Moreover, increasing the TOC concentration resulted in an increase of less bromated THMs.

The percentages of chloroform and bromated THMs in the TTHMs are given in Fig. 2. The TOC/Br$^-$ ratio influenced the relative levels of chloroform and the brominated THMs. When the concentration of bromide ions was increased, more brominated THMs were generated. An opposite tendency

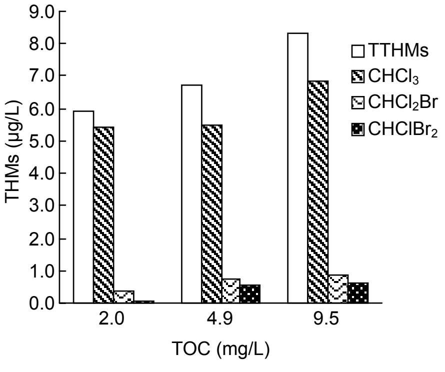

Fig. 1 Effect of the TOC concentration on the yields of THMs (reaction conditions: C_{Br^-}=3 mg/L, pH=7.5, time=24 h, temperature=25 °C, NH$_2$Cl dose=1 mg/L, Cl:N ratio=3:1)

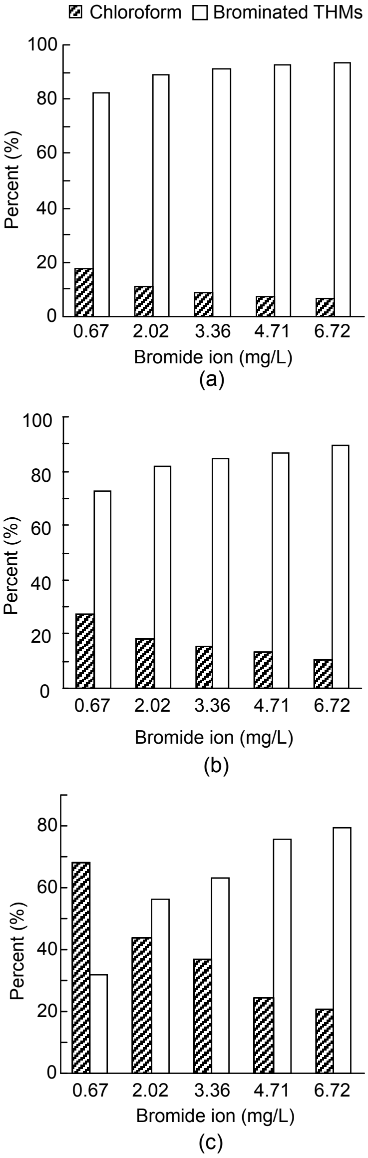

Fig. 2 Effect of the TOC concentration and bromide ion concentration on the percentages of chloroform and brominated THMs (reaction conditions: pH=7.5, time=24 h, temperature=25 °C, Cl$_2$ dose=2 mg/L)

(a) TOC=2.0 mg/L; (b) TOC=4.9 mg/L; (c) TOC=9.5 mg/L was found at the three TOC levels (2.0, 4.9, and 9.5 mg/L), in that the level of the dominant chloroform species dropped and that of brominated THMs increased. The increased TOC levels led to higher THMs formation and a higher proportion of brominated THMs, which means that more bromide was demanded for the brominated species. Even at the lowest concentration of bromide, a significant fraction of THMs was brominated species, reflecting the high reactivity of HOBr.

3.2 Effect of bromide ions on the formation of THMs

Fig. 3 illustrates THMs formation at various concentrations of bromide. There was some increase in the TTHMs concentration with increased bromide. This trend was very similar to that found in earlier research (Clark and Sivaganesan, 1998; Diehl *et al.*, 2000; Hua *et al.*, 2006; Yang *et al.*, 2007).

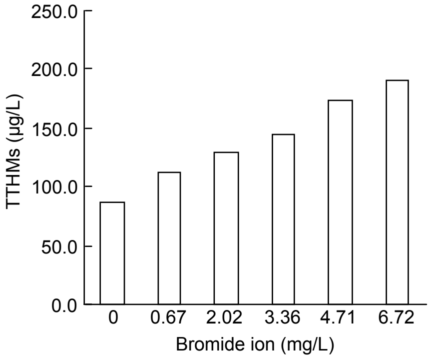

Fig. 3 Effect of bromide concentration on the yields of TTHMs (reaction conditions: pH=7.0, temperature=25 °C, time=24 h, TOC=5 mg/L, Cl_2 dose=2 mg/L)

Fig. 4 shows the formations of $CHCl_3$, $CHCl_2Br$, $CHClBr_2$, and $CHBr_3$ during chlorination with and without bromide spiking. The molecular weight of a bromide ion is 79.9 g/mol while that of a chloride ion is 35.5 g/mol. With a low concentration of added bromide ions (0.67 mg/L), part of the chloroform was replaced by brominated THMs, and the concentration of TTHMs increased from 87 to 112 μg/L (Fig. 3). There was a small change in the concentrations of the four THMs ($CHCl_3$, $CHCl_2Br$, $CHClBr_2$, and $CHBr_3$) when a low bromide concentration (0.67 mg/L) was added. The $CHCl_3$ concentration decreased gradually when the bromide concentration was increased to 6.72 mg/L. The $CHBr_3$ concentration increased to nearly 87 μg/L with increasing bromide concentration. The concentration of $CHClBr_2$ is more than tripled when the bromide concentration was increased from 0.67 to 2.02 mg/L, then dropped to 50 μg/L and finally reached a plateau with the increase of bromide concentration. The pattern of variation in $CHCl_2Br$ was almost identical to that of $CHClBr_2$ for the various levels of bromide ions. Of the four THMs, the most populous species was $CHBr_3$, and the least populous were $CHCl_3$ and $CHCl_2Br$.

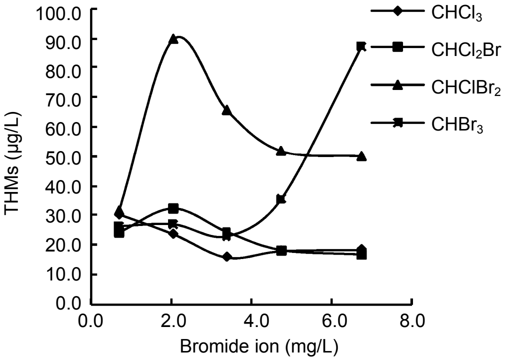

Fig. 4 Effect of bromide concentration on the formation and distribution of THMs (reaction conditions: pH=7.5, temperature=25 °C, time=24 h, TOC=5 mg/L, Cl_2 dose=2 mg/L)

The addition of bromide ions resulted in the formation of more bromine-containing THMs and a decrease in the formation of chlorine-containing THMs. The analysis clearly showed that the bromide ion concentration is the key element in the formation of THMs in chlorinated drinking water. A similar pattern also appeared at initial chlorine concentrations of 3, 5, and 10 mg/L. A model developed by Clark (1998) also showed that the presence of high levels of bromide ions supports the formation of brominated compounds. Yang *et al.* (2007) found the same tendency using the Suwannee River NOM.

Symons *et al.* (1993) recognized that the Br^-/HOCl ratio contributed to bromine substitution during THMs formation. At an added bromide ion level of 0.67 mg/L, the initial Cl/Br molar ratio of 2.9 led to a $CHCl_3/CHBr_3$ molar ratio of 1.2 and a $CHCl_2Br/CHClBr_2$ ratio of 0.8. At an added bromide ion level of 6.72 mg/L, the molar ratios of both $CHCl_3/CHBr_3$ and $CHCl_2Br/CHClBr_2$ were 0.3, which corresponded to an initial Cl:Br ratio of 0.3.

These data indicate that the reactivity of bromide ions with NOM is faster than that of chlorine and gives priority to bromine-containing THMs as the bromide ion concentration is increased. Westerhoff *et al.* (2004) and Hua *et al.* (2006) also showed that bromine substitution with NOM was about an order of magnitude faster than chlorine substitution. In a kinetics study, Krasner *et al.* (2006) investigated DBPs occurrence at 12 drinking water treatment plants. Their work indicated that the presence of bromide ions led to a transformation from chlorine-containing to bromine-containing DBPs. These results are consistent with those from our study.

3.3 Effect of the Cl:N ratio on the formation of THMs

Additional experiments were carried out to identify the effect of changing the Cl:N ratio on the formation of THMs. The selected Cl:N ratios were 3:1 and 5:1. Increasing the Cl:N ratio resulted in substantially increased concentrations of $CHCl_3$, $CHCl_2Br$, and $CHClBr_2$ (Fig. 5). The percentage of $CHCl_3$ dominated among the three THMs, amounting to 86% and 83% at Cl:N ratios of 3:1 and 5:1, respectively. The other two THMs ($CHCl_2Br$ and $CHClBr_2$) accounted for only 10% and 14%, and 3.9% and 3%, respectively. In practice, high levels of bromide ions are rare. These findings suggest that controlling the Cl:N ratio at a certain monochloramine level, could be a useful measure to reduce the production of $CHCl_3$. Similar patterns were also observed at initial chlorine concentrations of 2, 3, and 10 mg/L.

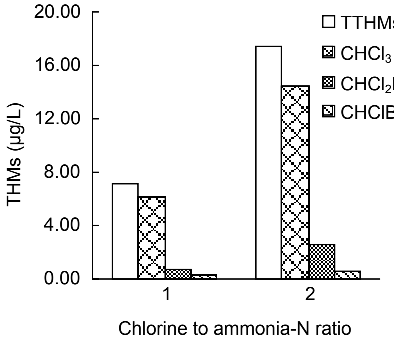

Fig. 5 Effect of the chlorine to ammonia-N ratio on THMs formation (reaction conditions: pH=7.5, temperature=25 °C, time=24 h, TOC=2.0 mg/L, C_{Br^-}=2.0 mg/L, NH_2Cl dose=5.0 mg/L)

3.4 Effect of the chloramine dose on the formation of THMs

Fig. 6 shows the formation of THMs after dosing with various monochloramine concentrations. A linear increase is observed in the TTHMs concentration as a function of the chloramination dose with different Cl:N ratios. According to the Pearson test, a strong relationship exists between the concentrations of THMs and the chloramine dose. The increments in TTHMs production per unit monochloramine dose were 0.45 and 1.54 mg/mg with Cl:N ratios of 3:1 and 5:1, respectively. THMs production was less affected by a monotonously increased monochloramine dose at a Cl:N ratio of 3:1, indicating that the Cl:N ratio is a key factor in controlling the production of THMs. These findings suggest that controlling the Cl:N ratio is also a good strategy to prevent increased THMs yields.

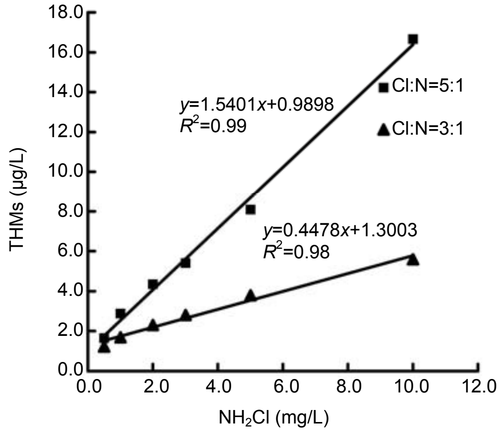

Fig. 6 THMs formation as a function of the chloramine dose (reaction conditions: pH=7.5, temperature=25 °C, time=24 h, TOC=2.0 mg/L, C_{Br^-} =0.6 mg/L, Cl:N=3:1 or 5:1)

3.5 Effect of bromide ion concentration on the decay of chloramines

Many studies have revealed that NOM and bromide ions can accelerate the decomposition of chloramines. Harrington *et al.* (2003) observed that the loss of chloramines occurred principally during the first 24 to 36 h of reaction time. Without bromide ions added, the monochloramine decay is caused mainly by chloramine autodecomposition and the presence of organic matter. Autodecomposition is a specific property of chloramine chemistry, and appears to be independent of the reactive components, such as bromide ions or organic matter. Fig. 7 shows the decay profile of monochloramine with and without bromide ion spiked. The monochloramine decay shows two reaction stages: a rapid initial decay followed by slower decay. In the first stage, the rapid decay is attributed mainly to active matter present in the water. When the bromide ion concentration of 0.67 mg/L was spiked, the monochloramine decay rates might have been caused in part by the presence of the bromide ions. These results reflect competitive reactions. The rate constant for the reaction between a bromide ion and hypochlorous acid (HOCl) is 1.55×10^3 $(\text{mol·s/L})^{-1}$ (reaction (1)) which is much higher than that for reactions (2) and (3). The following are some crucial reactions involving the decay of monochloramine:

$$\text{HOCl+Br}^{-}\rightarrow\text{HOBr+Cl}^{-}, \quad (1)$$

$k_1=1.55\times10^3$ $(\text{mol·s/L})^{-1}$ (Kumar and Margerum, 1987),

$$\text{HOCl+NOM}\rightarrow\text{Products}, \quad (2)$$

$k_2=(0.7\text{–}5.0)$ $(\text{mol·s/L})^{-1}$ (Westerhoff *et al.*, 2004),

$$\text{HOBr+NOM}\rightarrow\text{Products}, \quad (3)$$

$k_3=(15\text{–}167)$ $(\text{mol·s/L})^{-1}$ (Westerhoff *et al.*, 2004).

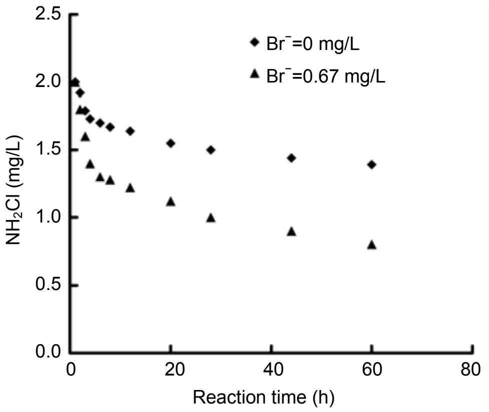

Fig. 7 Effect of the bromide ion concentration on chloramine decay (reaction conditions: pH=7.5, temperature=25 °C, time=24 h, TOC=5 mg/L, NH_2Cl dose=2.0 mg/L, Cl:N ratio=3:1)

4 Conclusions

The patterns of THMs formation found in this study are significant. The presence of bromide ions and TOC concentrations influenced the level and species of THMs formed during chlorination. The higher the TOC concentration, the higher the TTHMs produced.

At higher bromide ion concentrations, the generation of more bromated THMs was higher and the generation of less bromated THMs was lower. If raw water has high concentrations of bromide ions, relatively high levels of bromated THMs will be generated. Bromated THMs are more toxic than chlorinated THMs, so water utilities should pay more attention to the bromide ion level when using chloramination. In addition, bromide ions consume monochloramine, which increases the risk of microbial contamination.

Because the production of THMs is in direct proportion to the chloramine dose, the concentration of chloramines should be reduced to the lowest level feasible. Although disinfection is essential to prevent microbial contamination in drinking water distribution systems, its aftermath contributes to DBPs formation. It is important to evaluate and balance the microbial and chemical risks but, in practice, it is difficult to balance high disinfection efficiency with the occurrence of low levels of DBPs.

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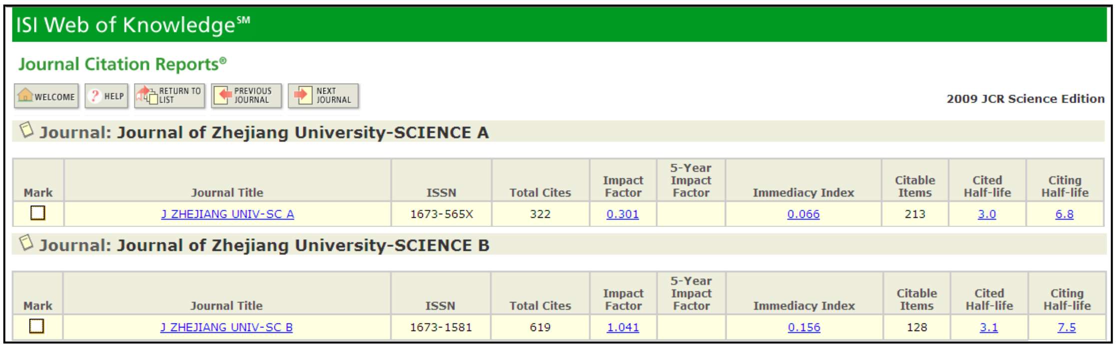

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Journal: Journal of Zhejiang University-SCIENCE B

Mark	Journal Title	ISSN	Total Cites	Impact Factor	5-Year Impact Factor	Immediacy Index	Citable Items	Cited Half-life	Citing Half-life
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