

# Rethinking aesthetic guidelines for manganese and iron in drinking water

A. E. Sain and A. M. Dietrich

## ABSTRACT

Manganese and iron are both internationally known causes of aesthetic issues in drinking water, however, there are limited data supporting their specific aesthetic guidelines, of which typical values are 0.05 mg/L Mn and 0.3 mg/L Fe. This study aims to clarify the concentrations at which off-flavors and off-colors caused by manganese and iron may be detected by consumers. Triangle tests of Mn(II) determined a best estimate taste threshold of 165 mg/L Mn(II), which is much higher than the reported range of 0.03–0.17 mg/L for Fe(II). Unlike Fe(II), Mn(II) taste tests showed there is no relationship between individual taste threshold and subject age. Mn(II) and Fe(II) oxidation in artificial saliva showed that Mn(II) had a non-detectable amount of oxidation and Fe(II) had up to 80% oxidation within 5 minutes at both 22 and 37 °C. Neither Mn(IV) nor Fe(III) exhibited detectable tastes. Visual testing of Mn(IV) and Fe(III), using the one-in-five forced choice method, showed that oxidized metals are visually detectable at concentrations below their typical aesthetic guidelines. Reduced Mn(II) and Fe(II) are colorless at concentrations much greater than established standards. This study demonstrates that current manganese and iron aesthetic standards may not be protective of off-flavors and off-colors.

**Key words** | color, flavor, iron, manganese, metallic, taste

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## INTRODUCTION

Manganese and iron are both metallic elements that are known to cause aesthetic issues in drinking water. Globally, both have been identified as sources of off-colors and off-flavors. Manganese and iron are both abundant in the Earth's crust, and therefore, both are often found in drinking water sources that come in contact with iron and manganese containing rock or sediment. Manganese concentrations between 0.1 and 1 mg/L and iron concentrations of 0.05–10 mg/L are common in source waters. In ground waters, which are low in oxygen, both iron and manganese can be present in their soluble, reduced forms, Fe(II) and Mn(II). This is also true of surface waters, which are low in oxygen, specifically in the hypolimnion of stratified lakes and reservoirs. Conversely, the oxidized forms of both iron (Fe(III)) and manganese (Mn(IV)) are poorly soluble in surface and ground waters

of sufficient dissolved oxygen or under oxidizing conditions. Owing to similarities in chemistry and occurrence, treatments to remove manganese and iron are also similar (Crittenden *et al.* 2012).

Internationally, manganese and iron have similar drinking water standards (Table 1). Currently, the World Health Organization (WHO) does not have established guidelines for either manganese or iron, but does acknowledge that aesthetic issues may occur (WHO 2011).

Treatment to remove manganese and iron is often implemented to limit their impact on the aesthetic quality of drinking water. The basis for current aesthetic guidelines is unclear, as is how protective they may be of off-colors and off-flavors. Manganese guidelines typically cite its 'metallic, bitter taste; black/brown color or staining' as aesthetic issues, while iron guidelines typically

**Table 1** | International standards for manganese and iron in drinking water

Organization	Type	Compliance <sup>f</sup>	Manganese	Iron
World Health Organization <sup>a</sup>	–	–	No value	No value
US Environmental Protection Agency <sup>b</sup>	Aesthetic	Not enforced	0.05 mg/L	0.3 mg/L
European Drinking Water Directive <sup>c</sup>	Aesthetic	Enforced <sup>g</sup>	50 µg/L	200 µg/L
Australia Drinking Water Guidelines <sup>d</sup>	Health	Enforced	0.5 mg/L	None
	Aesthetic	Not enforced	0.1 mg/L	0.3 mg/L
Taiwan Environmental Protection Administration <sup>e</sup>	Aesthetic	Enforced	0.05 mg/L	0.3 mg/L

<sup>a</sup>WHO (2011).<sup>b</sup>USEPA (2013).<sup>c</sup>EC 98/83/EC (1998).<sup>d</sup>NHMRC/ARMCANZ (2011).<sup>e</sup>ROC (Taiwan) Environmental Protection Administration (2009).<sup>f</sup>Required, enforced standard or, if optional, not enforced.<sup>g</sup>In cases where exceedance may result in a risk to human health as determined by member states.

cite ‘metallic taste; rust/red/brown color or staining’ (USEPA 2013). Previous studies established that the population taste threshold for iron is 0.03–0.17 mg/L Fe(II) and that age impacts ability to taste iron (Cohen *et al.* 1960; Mirlohi *et al.* 2011; Ömür-Özbek & Dietrich 2011). Fewer data are available regarding population taste thresholds for manganese. Sain *et al.* (2014) determined a Mn(II) taste threshold from 71 to 101 mg/L, and Cohen *et al.* (1960) determined a Mn(II) taste threshold of 45 mg/L; age was not examined as a factor. Salivary lipid oxidation contributes to the flavor of ferrous iron in drinking water (Ömür-Özbek *et al.* 2012), but there is limited information about salivary lipid oxidation of manganese. Sain *et al.* (2014) demonstrated that Mn(IV) imparts off-color to water at concentrations <10% of the USEPA secondary maximum contaminant level (SMCL) of 0.05 mg/L (USEPA 2013). Off-color is also attributed to iron in drinking water, but the concentration at which off-colors can be detected is not well explored. Objectives for this study include determination of age relationship for manganese taste threshold, determination of manganese and iron oxidation in saliva, and determination of concentrations resulting in visual detection of oxidized metals in drinking water. The more defined thresholds for off-colors and off-flavors caused by manganese and iron in drinking water will assist drinking water treatment professionals and consumers in ensuring their water is free from aesthetic issues.

## METHODS

### Manganese taste threshold

In accordance with Institutional Review Board Protocol # 12–710, 57 adults (21 female) performed a series of 10 taste tests for manganese concentrations ranging from 8.8 to 337.5 mg/L. Subjects were 18 or older, of any health status but not known to be pregnant, and abstained from food or beverages for 1 hour preceding testing. The taste tests occurred in ascending order over 1 hour. The approach was similar to ASTM (American Standards for Testing Materials) E679-04 (2011), which was appropriate as previous research (Sain *et al.* 2014) demonstrated that Mn(II) did not require long rest periods between samples as is necessary for Fe(II). Triangle taste tests utilized 30 mL solution in 5 oz. taste-and-odor free plastic cups (Solo Brand) and taste-and-odor free deionized Milli-Q<sup>®</sup> water as the control. Food grade MnSO<sub>4</sub> (Spectrum, CAS 10034-96-5) was dissolved in deionized water to prepare concentrations from 8.8 to 337.5 mg/L; a 1.5× multiplier was used between concentrations. Concentrations were confirmed by atomic absorption spectroscopy (Perkin Elmer Model 5100) or Thermo Electron X-Series inductively coupled plasma with mass spectrometer. Individual taste thresholds were calculated by taking the geometric mean of the highest missed concentration and lowest correctly identified concentration after three sequential concentrations were correctly identified. If the highest

concentration given (337.5 mg/L Mn(II)) was not correctly identified the individual threshold was calculated as if the next concentration using the 1.5× multiplier would have been correctly identified (506.3 mg/L Mn(II)). A best estimate threshold (BET) for the population was calculated as the geometric mean of all individual taste thresholds.

## Metal oxidation in artificial saliva

### Artificial saliva

Artificial inorganic saliva was prepared to contain 752 mg/L  $\text{Cl}^-$ , 51 mg/L  $\text{SCN}^-$ , 378 mg/L  $\text{HCO}_3^-$ , 770 mg/L  $\text{K}^+$ , 303 mg/L  $\text{Na}^+$ , 60 mg/L  $\text{NH}_4$ , 457 mg/L  $\text{PO}_4^{3-}$ , and 228 mg/L  $\text{SO}_4^{2-}$  as described in [Hong \*et al.\* \(2006\)](#) and [Gal \*et al.\* \(2001\)](#). To create the lipid-amended artificial saliva, 0.030 g of soybean oil (Spectrum, CAS 8001-22-7) was added per 100 mL of artificial saliva mixture.

### Speciation of manganese

A solution of 0.04% leucoberbeline blue dye (Sigma-Aldrich, CAS 52748-86-4) in 45 mM acetic acid (Fisher Scientific, CAS 64-19-7) was used to determine oxidized Mn (Mn(III), (IV), or (VII)) of which Mn(IV) is the most likely oxidation product of Mn(II) ([Boogerd & de Vrind 1987](#)). Standards, prepared in the range of 0.05–10 mg/L Mn from  $\text{KMnO}_4$  (Fisher Scientific, CAS 7722-64-7), at both 22 and 37 °C (body temperature) were measured at 620 nm as recommended in [Boogerd & de Vrind \(1987\)](#). The USEPA limit of detection method ([Keith \*et al.\* 1983](#)) was used to calculate a limit of detection for the leucoberbeline blue method. It was determined that the limit of detection is an absorbance value of 0.054 with a 1 mg/L  $\text{KMnO}_4$  standard used to determine standard deviation.

### Speciation of iron

The phenanthroline method ([Standard Methods for the Examination of Water and Wastewater 2006](#)) was used to determine Fe(II) in solutions through formation of a phenanthroline-ferrous complex with an absorbance maxima at 510 nm. The phenanthroline method measures ferrous iron, so a decrease in ferrous iron indicates conversion to ferric iron.

## Reaction of reduced metal with artificial saliva

Fe(II) or Mn(II) solutions were made by the addition of 5 mL of  $\text{FeSO}_4$  (Spectrum, CAS 7782-63-0) or  $\text{MnSO}_4$  stock solution to 45 mL of the desired media (deionized water, artificial saliva, and lipid-amended artificial saliva) with a resulting concentration of 10 mg/L Fe(II) (180 mM Fe(II)) or 9.84 mg/L as Mn(II) (180 mM Mn(II)). After reaction for 1, 2, 5, and 60 minutes, Fe(II) was measured by the phenanthroline method or Mn(IV) was measured by the leucoberbeline method. Reaction times of 1–5 minutes represent times a sip of water may stay in a person's mouth, while 60 minutes was an extended time period. Experiments were performed in triplicate at 22 and 37 °C (body temperature). A multi-way analysis of variance (ANOVA) performed in R 3.0.1 ([R Development Core Team 2013](#)) was utilized to measure deviations between treatments within the manganese or iron experiments.

### Visual testing for iron

The one-in-five forced choice method was used to determine the visual perception of iron in drinking water. Solutions of  $\text{FeSO}_4$  dissolved in deionized water at room temperature and iron concentrations at 100, 60, 20, and 10% of the SMCL (0.3 mg/L Fe) and then oxidized in the laboratory by the addition of approximately 0.2 mL of 6% NaOCl per 250 mL of sample solution. A 100% SMCL sample was also prepared in deionized water immediately before visual testing to provide an unoxidized ferrous iron sample for comparison. Volumes of 250 mL of each solution were poured into semi-opaque plastic cups, which were randomly placed with four 250 mL deionized water controls. This method was applied previously to assess the visual characteristics of reduced and oxidized manganese ([Sain \*et al.\* 2014](#)).

## RESULTS

### Manganese taste thresholds

The results of the Mn(II) triangle taste tests can be seen in [Figure 1](#). Individual taste thresholds for the 57 subjects

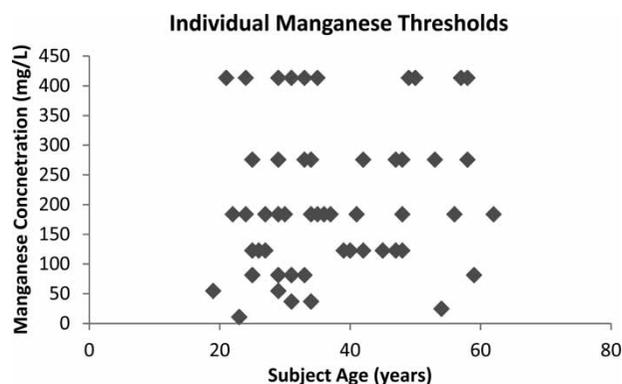


Figure 1 | Individual Mn(II) taste thresholds for  $n = 57$  subjects.

ranged from 10.7 to 413.3 mg/L Mn(II). The calculated 50% BET for the population is 165 mg/L Mn(II). There was no correlation found between age and individual taste threshold of subjects ( $R^2 = 0.0087$ ).

### Metal oxidation in artificial saliva

#### Manganous manganese

The leucoberbeline blue method cannot detect oxidized manganese at absorbances lower than 0.054, which was equivalent to 1.23 mg/L Mn according to a standard curve with measurements of 0, 3, 6, 9, and 12 mg/L  $\text{KMnO}_4$  (Keith *et al.* 1983). Using this limit of detection, no detectable amount of Mn(II) oxidation was observed at 1, 2, or 5 minutes at either 22 or 37 °C for initial concentrations of 9.84 mg/L Mn(II) (180 mM Mn(II)) in artificial saliva, lipid-amended artificial saliva, or deionized water. Samples taken after 60 minutes for each media and temperature condition contained visible precipitation, which confounded results.

#### Ferrous iron

The initial concentration of ferrous iron in all solutions was 10 mg/L (180 mM Fe(II)). Results show that for inorganic saliva and lipid-amended inorganic saliva, there is a significant ( $P < 0.05$ ) reduction in ferrous iron concentration at both 22 and 37 °C and the majority of the reactivity occurs within 1 minute then continues over time (Figure 2). There was not a significant amount of oxidation over time for

ferrous iron in deionized water at either 22 or 37 °C. For both inorganic saliva and lipid-amended inorganic saliva, there is a significant difference between ferrous iron concentrations at 22 and 37 °C at each time point. There was no significant difference between inorganic saliva and lipid-amended inorganic saliva at each time point for each temperature, but both saliva types were significantly different from the deionized water solution at each time for each temperature.

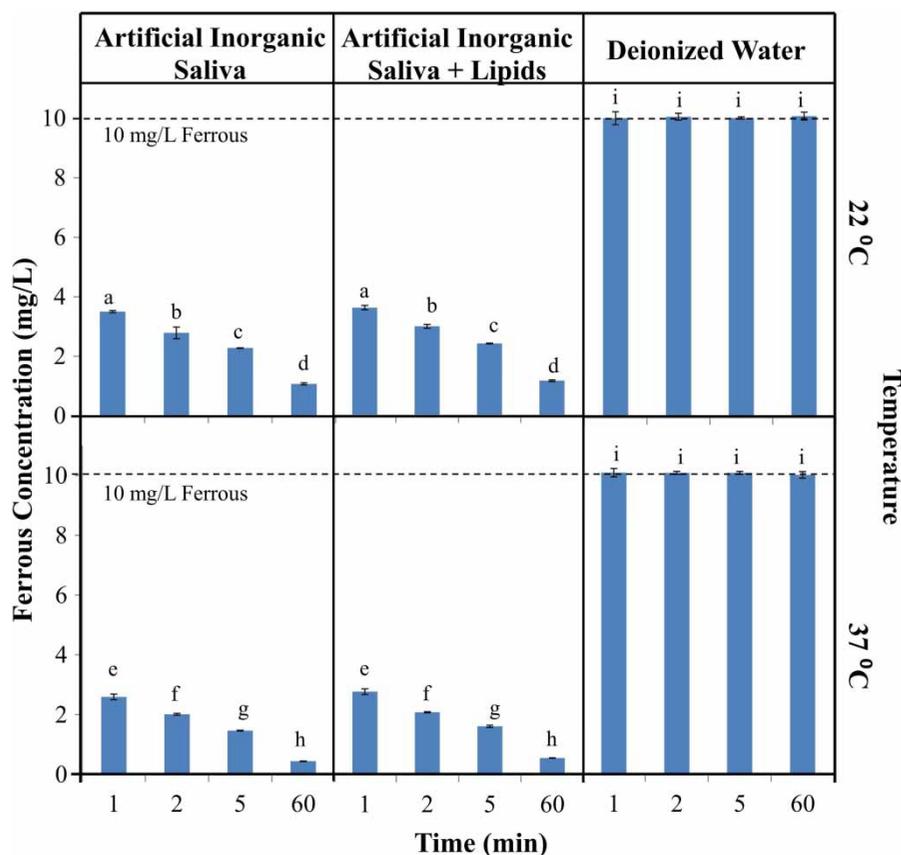
### Visual detection of oxidized iron

Using a forced choice one-in-five test, oxidized ferric iron concentrations at 0.3, 0.18, 0.06, and 0.03 mg/L were viewed with deionized water as the control. The oxidized iron samples at concentrations at and below the USEPA SMCL (0.3 mg/L Fe(III)) were apparent as a yellow-orange discoloration. The percentages of subjects detecting the oxidized iron at each concentration are presented in Table 2. The percentages indicate 97% detection at 0.3 mg/L Fe(III) decreasing to 21% at 0.03 mg/L. Manganese data from a previous study at the same percentages of the SMCL are included in Table 2 (Sain *et al.* 2014). Neither Mn(II) nor Fe(II) had detectable color at their respective SMCLs.

## DISCUSSION

### Manganese and iron flavor perception

The Mn(II) taste threshold was determined to be 165 mg/L Mn(II) by the BET method in this research. Previously reported thresholds varied from 45 mg/L Mn(II) (Cohen *et al.* 1960) and 75 mg/L Mn(II) (logistic regression method) to 101 mg/L Mn(II) (BET method) (Sain *et al.* 2014). The threshold reported here is slightly higher than those previously reported, all of which used different test methods to determine Mn(II) thresholds. The test subjects, methods, and conditions are all acknowledged to affect a measured threshold as 'a threshold is not a constant for a given substance, but rather a constantly changing point on the sensory continuum from non-perceptible to easily perceptible' (Meilgaard *et al.* 1987).



**Figure 2** | Ferrous iron oxidation over time in artificial salivas and deionized water. Error bars show 95% confidence interval; a–i signify groups that are not significantly different ( $P > 0.05$ ).

**Table 2** | Percentage of subjects detecting oxidized iron and oxidized manganese in solution

Percent of SMCL <sup>a</sup>	Percent of subjects positively detecting	
	Oxidized manganese <sup>b</sup> Mn(IV) (n = 31)	Oxidized iron Fe(III) (n = 64)
100	100	97
60	97	84
20	97	50
10	97	21 <sup>c</sup>

<sup>a</sup>USEPA SMCL = 0.3 mg/L Fe; USEPA SMCL = 0.05 mg/L Mn (USEPA).

<sup>b</sup>Sain *et al.* (2014).

<sup>c</sup>Equal to chance.

There was little evidence for oxidation of Mn(II) in saliva. Within the detection limits of the leucoberbeline method, less than ~10% Mn(II), and perhaps no Mn(II), was oxidized within the 5-minute reaction period with artificial saliva with or without lipids. This is in contrast to Fe(II), where nearly 80% of the Fe(II) was consumed and likely

oxidized to Fe(III) in the 5-minute reaction time with artificial saliva. Previous research demonstrated that organic molecules associated with salivary lipid oxidation could not be measured for Mn(II) (Sain *et al.* 2014), but were readily measured for Fe(II) (Ömür-Özbek *et al.* 2012). Salivary lipid oxidation was previously demonstrated to be a critical factor in the flavor of ferrous iron. Aqueous Fe(II) is well established to have flavor, with a retronasal metallic odor dominating over a weak taste sensation (Ömür-Özbek & Dietrich 2011; Mirlohi *et al.* 2011; Ömür-Özbek *et al.* 2012).

The Mn(II) population taste threshold is much greater – approximately 1,000-fold greater – than the Fe(II) iron population flavor threshold, previously established to be 0.03–0.17 mg/L Fe(II) (Mirlohi *et al.* 2011; Ömür-Özbek & Dietrich 2011) (1,000-fold is based on estimating thresholds of 100 mg/L Mn and 0.1 mg/L Fe). Age is known to impact ability for sensing iron. Population thresholds for people <50 and >50 years are 0.045 and 0.498 mg/L Fe(II), respectively (Mirlohi *et al.* 2011). The typical oxidized forms

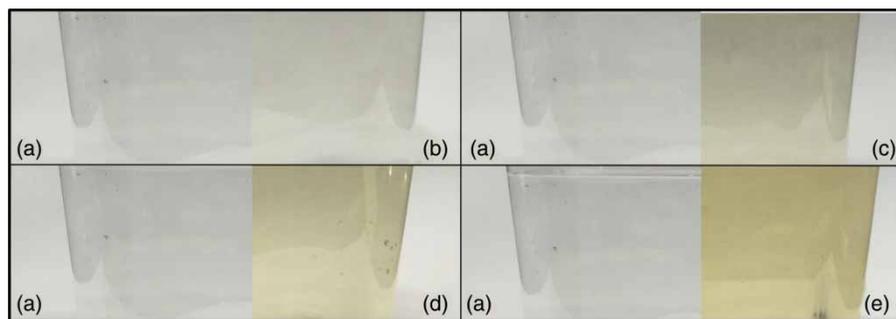
of these metals in aqueous solution have no flavor sensation. Manganic manganese, Mn(IV), was shown to have no detectable taste at the SMCL = 0.05 mg/L (Sain *et al.* 2014), and ferric iron has been shown to not have detectable metallic taste or odor and not to cause lipid oxidation (Ömür-Özbek *et al.* 2012).

Examination of individual Mn(II) taste thresholds reveals there is variability in ability to taste Mn(II) within the population. Taste thresholds vary by more than 400 mg/L between individuals. This study showed that unlike iron, there is no relationship between age and the ability to detect manganese taste in drinking water. One possible explanation for the variability in calculated taste thresholds is the potential for the results to be impacted by total dissolved solids (TDS) effects. The TDS concentrations at the calculated thresholds would be 206–454 mg/L TDS, which is above the 85 mg/L TDS consumers were found to prefer compared to distilled water (Bruvold 1968), and below the USEPA and Taiwanese aesthetic guideline of 500 mg/L TDS (2013). It is clear that the taste threshold for manganese is far higher than current manganese guidelines (Table 1), and therefore, current regulations should be considered to be protective of off-flavors from manganese. However, it is important to note that the high manganese taste threshold indicates that consumers would most likely not be able to detect elevated manganese concentrations in their drinking water, which could be harmful to human health, especially in children (Bouchard *et al.* 2007, 2011; Khan *et al.* 2011). There is a clear contrast between the impact of reduced iron and reduced manganese on the flavor of a drinking

water, with iron having the potential to adversely impact flavor of drinking water at concentrations below the current international aesthetic guidelines and sensory ability impacted by age, and manganese having little effect on the flavor of drinking water at concentrations near and even far greater than the current international aesthetic guidelines with no relationship between tasting ability and subjects' age.

### Manganese and iron visual perception

To more fully examine the aesthetic differences between iron and manganese, tests to determine the ability to visually detect oxidized iron were performed. The results suggest that many consumers are able to detect an off-color due to Fe(III) at the USEPA SMCL (2013), 0.3 mg/L Fe. This standard is widely used in the international community (Table 1) with the exception of in the EU, and may not be sufficient for protecting against off-color in drinking water under all drinking water conditions of different glass sizes, colors, and water volumes. The 200 µg/L standard recommended by the EU (EC 98/83/EC 1998) is also likely not protective of off-color as 84% of subjects in this study were able to detect 0.18 mg/L, or 60% of the USEPA SMCL (2013). At 10% of the USEPA SMCL (2013), 0.03 mg/L, the percentage of the population that detected iron positively (21%) has dropped to a level that is consistent with the chance of guessing the correct sample (1 of 5 or 20%). Similar examination of manganese has shown that the current aesthetic standards are even less effective at protecting against off-color than the iron



**Figure 3** | Visual effects of oxidized Mn(IV) in water at varying concentration. Images show a detectable off-color when Mn(IV) was added to  $L3.5' \times W3.5' \times H4'$  containers with 500 mL of (a) deionized Milli-Q® water, (b) 0.05 mg/L Mn(IV), (c) 0.1 mg/L Mn(IV), (d) 0.3 mg/L Mn(IV), and (e) 0.5 mg/L Mn(IV). Mn(IV) was prepared by oxidizing  $Mn^{2+}$  with chlorine to form  $MnO_2$ .

**Table 3** | Aesthetic characteristics of reduced and oxidized manganese and iron

Characteristic Typical aesthetic guideline Taste/Flavor	Manganese		Iron	
	0.05 mg/L Mn		0.3 mg/L Fe	
	Mn(II)	Mn(IV)	Fe(II)	Fe(III)
Threshold	75–165 mg/L	NA	0.03–0.17 mg/L	NA
Salivary lipid oxidation	No	NA <sup>a</sup>	Yes	NA
Retronasal odor	No	NA	Yes	No <sup>b</sup>
Age trend	No	NA	Yes	NA
TDS contribution	Yes	NA	No	NA
Visual detection				
Aesthetic guideline	No	Yes	No	Yes
10% aesthetic guideline	No	Yes	No	No

<sup>a</sup>NA: not analyzed as this metal species has no substantial taste/Flavor.

<sup>b</sup>Ömür-Özbek & Dietrich (2011).

standards as 97% of people are able to detect oxidized manganese at 10% of the current USEPA SMCL, which is shared by much of the international community (Table 1) (Sain *et al.* 2014). Examples of oxidized manganese in water are available in Figure 3. These data suggest that in order to protect against off-colors, consumer complaints, and maintain consumer confidence in water safety, utilities should achieve maximum removal of iron and manganese.

## CONCLUSIONS

Although manganese and iron are often treated similarly in engineering guidelines, there are clear aesthetic differences between the two metals and their oxidation states, as summarized in Table 3. Elevated Fe(II) concentrations are much more likely than Mn(II) to result in an off-flavor, even at concentrations less than international aesthetic guidelines. There is no evidence that Mn(II) or Mn(IV) exhibits a bitter or metallic taste at the 0.05–0.1 mg/L Mn concentrations commonly applied for aesthetic standards. In contrast, Mn(IV) is more likely than Fe(III) to result in detection of an off-color, but both metals can be detected at and below the current aesthetic guidelines. In conclusion, utilities and homeowners should make efforts to minimize both manganese and iron in drinking waters to protect against off-flavors and colors.

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